EVALUATION OF VACUUM PACKAGING ON THE PHYSICAL PROPERTIES, SOLUBILITY, AND STORAGE SPACE OF DAIRY POWDERS

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ABSTRACT

EVALUATION OF VACUUM PACKAGING ON THE PHYSICAL PROPERTIES, SOLUBILITY, AND STORAGE SPACE OF DAIRY POWDERS

Hadi Eshpari

As many of the dairy powders manufactured have to travel long distances to reach their customers, both domestically and internationally, there is considerable interest among dairy powder manufacturers to maintain the quality of their products for relatively long storage periods. Dairy powders can have a long shelf life if packaged and stored properly. Vacuum packaging can be an attractive packaging strategy to maintain the quality of dairy powders and provide added value by improving the efficiency of using the storage space; because of the inherent compactness of these products. Vacuum packaged dry dairy ingredients may also have added ease of handling for end users. However, little is known about the impact of vacuum packaging on the physical properties of dry dairy ingredients. The main objective of this study was to determine the effect of vacuum packaging over 12 months storage on particle size, particle density, bulk density, tapped density, flowability, compressibility, color, moisture content, surface morphology, and solubility of six types of dairy powders. In addition, the effect of dairy ingredients type was also assessed. Commercial samples of nonfat dry milk powder, whole milk powder, buttermilk powder, milk protein Isolate, whey protein concentrate#80, and sweet whey powder were repackaged in duplicate using multi-wall foil side gusseted bags under varying degrees of vacuum (1, 0.7, 0.4 bar) and a control with no vacuum, then stored for 3, 6, and 12 months at 25°C and 60% relative humidity.

Each powder was sampled and analyzed in duplicate for all the above listed quality attributes, upon receiving the powder and after 3, 6, and 12 months of storage.

Moreover, the effect of vacuum packaging on storage space was evaluated comparing three different models; Model (1) represented a 25 kg bag of atmospheric packaged non fat dry milk with the actual dimensions of a commercial 25 kg bag of non fat dry milk. Model (2), a hypothetical model, represented a 25 kg bag of vacuum packaged non fat dry milk with a length and a width equal to those of model (1). Model (3), another hypothetical model, also represented a 25 kg bag of vacuum packaged non fat dry milk with a length equal to half of a pallet width and a width equal to one third of a pallet length, in order to achieve the highest pallet efficiency possible. The pallet used for all three models was considered to be a (48 × 40) pallet. The height of models 2 and 3 was allowed to reflect the bulk reduction effect of vacuum packaging and was determined based on the weight, density and the known dimensions of the bags. It is important to note that the density of models 2 and 3 was assumed to be equal to the density of a small bag of nonfat dry milk. The saved space per bag and pallet efficiency of vacuum packaging and atmospheric packaging were compared using the three models described above.

Physical properties analyses of the dairy powders revealed statistically significant effect of vacuum pressure on only color values: L-, a-, and b but none of the other powder quality attributes examined. Powders packaged under vacuum showed a significantly higher mean of L- color value (p-value = 0.003 < 0.01), but significantly lower means of (a- and b-) color values (p-values = 0.005, and 0.001, respectively). This effect was more dramatic in high fat containing powder such as whole milk powder. In

fact, vacuum packaged whole milk powders were significantly whiter, less red, and less yellow. It is likely that vacuum packaging has prevented color changes due to lipid oxidation in whole milk powder.

Physical properties analyses of the dairy powders also revealed statistically significant increases in the particle density, particle size, bulk density, and tapped density due to the effect of storage time (all p-values = 0.000 < 0.01), statistically significant decreases in the angle of repose and compressibility due to the effect of storage time (p = 0.000 < 0.01) and (p = 0.004 < 0.01), respectively. The physical properties analyses also revealed a statistically significant effect of the powder type on particle density, particle size, bulk density, and tapped density, angle of repose, compressibility, and color values: L-, a-, and b- (all p-values = 0.000 < 0.01). In other words, particle density, particle size, bulk density, and tapped density of the powders increased over the storage time, while angle of repose (AOR) and compressibility decreased over the storage time. The powder type had a significant effect on particle density, particle size, bulk density, tapped density, AOR, compressibility, and color values: L-, a-, and b; however, it did not have any significant effect on solubility and moisture content.

In addition, observations of the surface morphology of dairy powders were made using a scanning electron microscope. This evaluation demonstrated the differences in powder particle shape and surface morphology which are believed to be partially responsible for the significant differences observed in the physical properties, due to the effect of powder type.

It was shown that vacuum packaging does increase the efficiency of using the storage space by removing the interstitial air and increasing the density of the powder.

As described above, the height of model (2) and the length of model (3) both were expectedly shorter compared to those of model (1). Storage space calculations for non fat dry milk were performed based on comparing the volume of the 3 models and showed 15 % saving in storage space per bag and per pallet, due to vacuum packaging. The effect of space saving on the number of bags per pallet was evaluated using CAPE PACK v2.09 software and showed an increase from 45 bags/ pallet in model (1) to 50 bags/ pallet in model (2) and 54 bags/ pallet in model (3).

Overall, this study demonstrates the impact of vacuum packaging on physical properties, solubility, and storage properties of dairy powders. The data suggest that the proposed vacuum packaging method may be beneficial to maintain the quality of the powders studied and it results in space savings per unit of dairy powder compared to conventional atmospheric packaging.

Keywords: vacuum packaging, physical properties, solubility, storage properties, dairy powders

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1. INTRODUCTION

The atmosphere surrounding the food in a package has a profound effect on the shelf life of the product. One of the principal techniques that makes use of the in package atmosphere for improving the preserving action of the package is vacuum packaging. Vacuum packaging is a method of storing food and presenting it for sale. It basically refers to packaging in containers (rigid or flexible), from which some or all the air has been removed prior to final sealing of the container. The objective of removing air and specifically the atmospheric oxygen is to prevent oxidation reactions such as lipid oxidation, oxidative browning, loss of certain vitamins etc. Vacuum also prevents deterioration by aerobic microorganisms and particularly mold. Vacuum packaging is commonly used for long-term storage of dry foods such as cereals, nuts, cured meats, cheese, smoked fish, coffee, and crisps. It also offers the additional advantages of reducing the volume (bulk) of the food. This may be important for an efficient use of space in the storage and transportation of food products.

As a result of globalization, dry dairy ingredients are among the food items that are being exported from leading dairy producing countries to countries with limited milk supply, most of which have adverse climatic conditions of high temperature and high relative humidity. Consequently, dry dairy ingredients can have a long shelf life in such harsh storage conditions, only if packaged and stored properly.

Vacuum packaging may be an attractive method to dairy powder industry, for keeping quality and its potential of providing added value by improving ease of handling for end users. As the demand for maintaining the quality of dry dairy ingredients increases, researchers are striving to understand and improve the packaging

characteristics influencing the quality of dry dairy ingredients during the storage time. However, little is known about the impact of vacuum packaging on the physical properties of dry dairy ingredients. It was hypothesized that (1) Vacuum packaging does not negatively impact physical properties of dairy powders. (2) Vacuum packaging does save space in storage by reducing the bulk density of dairy powders.

Therefore the goal of this study was to determine if vacuum packaging may result in dry dairy ingredients with improved quality and higher efficiency of using the storage space. More specifically, the objective was to determine the effects of vacuum packaging and storage on physical properties and solubility of dry dairy ingredients and finally to provide directions for further investigations of packaging methods that not only maintain the quality of dry dairy ingredients but also improve their handling properties, stacking stability, and the efficiency of using the space in storage and transportation.

2. LITERATURE REVIEW

2.1. Brief Outline

This literature review aims to establish a common foundation of knowledge in the area of dry dairy ingredients packaging with respect to its impact on the physical properties and solubility of the powders during the storage. This literature review will first define select dairy powders and then examine the current method of packaging dry dairy powders available domestically, while paying special consideration to correlations between the packaging characteristics and the changes in the quality attributes of dairy powders over the storage time. Some packaging related problems observed in handling and storage of dairy powders will be discussed, along with giving a definition of different powder properties studied in this project. Finally, an experiment will be proposed to more thoroughly investigate the effects of vacuum packaging on the physical properties and solubility of dry dairy powders, during a 12 month storage period.

2.2. Significance of Packaging

Dairy powders are immensely more stable than fresh milk but protection from moisture, oxygen, light and heat is needed in order to maintain their quality and shelf life. Dairy powders readily take up moisture from the air, leading to a rapid loss of their quality (Fitzpatrick et al., 2007). Dairy powders with high fat content such as whole milk powder tend to develop off flavors during storage time. This is caused by the reaction of fat with oxygen in the air, especially at higher storage temperatures (> 30°C), typical of the tropical climates (Farkye et al, 2001). Therefore, packaging has to be chosen to provide a barrier to moisture, oxygen and light.

2.3. Modified Atmosphere Packaging

Modified atmosphere packaging (MAP) simply means packaging a food in an atmosphere that is different from the normal composition of air (78.08% N_2, 20.96% O_2, 0.03% CO_2, variable amounts of water and traces of inert gases) (Farber and Dodds, 1995). Under this general definition, several packaging techniques are included, such as vacuum packaging, controlled atmosphere and gas flush packaging. Vacuum packaging is perhaps the most common method of modifying the internal package atmosphere (Farber and Dodds, 1995), however it has not been used to any great extent for dairy powder packaging and its potential is yet to be thoroughly investigated.

2.4. Definition of Vacuum Packaging

In vacuum packaging, the product is placed in a package of low O_2 permeability, the air is fully or partially evacuated and the package is sealed without deliberate replacement with another gas mixture (Church, 1998).

2.4.1. Advantages of Vacuum Packaging

The two main advantages of vacuum packaging dairy powders are expected to be: (1) Extending shelf life; by minimizing or removing air from a package, the chemical and biological deterioration of food in the package are greatly inhibited, as both deteriorations are concerned with oxygen and oxidation of fat. Lloyd et al., (2009) showed that air-packaged WMP has higher peroxide values, lipid oxidation volatiles, and off flavors (grassy and painty) than nitrogen-flushed WMP. The same can be true about vacuum packaged dairy powders. Deteriorations such as discoloration and rancidity of food with aerobic microorganisms are typical (Farber and Dodds, 1995). In fact, before ambient air

begins to directly deteriorate (drying out, oxidation, denature of aromas) or indirectly deteriorate (favoring the development of bacteria, yeast, mould and their metabolisms) the food product, vacuum packaging can "freeze" the quality of the food product at the level reached in the production phase and maintain it intact up to the moment of consumption. (2) Minimizing storage space; package is drawn tight around the food and the food volume is reduced under the vacuum pressure, taking up minimal space. The intensity of the bulk reduction depends on the porosity and the amount of air in the dry dairy ingredient. Clearly, this effect would be significant for dairy powders with so many voids between and within the particles; however, there is a lack of literature showing this advantage of vacuum packaging quantitatively.

2.4.2. Disadvantages of Vacuum Packaging

The main disadvantage of vacuum packaging, especially for a production plant that is already equipped with other types of packaging machines, might be the cost of buying a vacuum packaging device. However, the advantages of vacuum packaging, such as increasing the efficiency of using storage space, packaging material, and eliminating the need of nitrogen gas, are expected to eliminate these disadvantages. Although the cost related aspects of vacuum packaging, conventional atmospheric packaging, and gas flush packaging are well worth studying.

2.5. Definition of Dried Dairy Powders

Dairy powders represent a diverse range of dairy products. They vary considerably in chemical composition, which is determined by the composition of the original milk as well as the various heating, dehydration and other processing steps

involved in their manufacture (Tamime, 2009). There is also variation in the distribution of chemical components within products, for example between the surface and the interior of powder particles and between the colloidal and soluble phases, which affects the products' properties (Tamime, 2009).

Chemical and enzymic changes continue to occur during storage of the products, which can significantly affect their functional properties and organoleptic qualities. The most important chemical changes that occur or can occur during processing and storage are denaturation of whey proteins, coagulation of caseins, lactosylation of proteins and subsequent Maillard reactions, oxidation of milk fat and crystallisation of lactose. Knowledge of the chemical components of the products, their relationship to functional properties, and the changes that can occur in these components is essential for determining the optimal packaging and storage conditions for these products (Tamime, 2009).

The select dry dairy ingredients for the purpose of this project which would be defined in the next sections, are; non fat dry milk (NFDM), whole milk powder (WMP), buttermilk powder (BMP), whey protein concentrate (WPC), milk protein isolate (MPI) and sweet whey powder (SWP).

2.5.1. Definition of Non-Fat Dry Milk & Skimmed Milk Powder

Nonfat dry milk and skimmed milk powder are very similar. They are manufactured by removing water from pasteurized skim milk. Both contain 5% or less moisture (by weight) and 1.5% or less milkfat (by weight). The difference is that skimmed milk powder has a minimum milk protein content of 34%, whereas nonfat dry milk has no standardized protein level (American Dairy Products Institute, 1990).

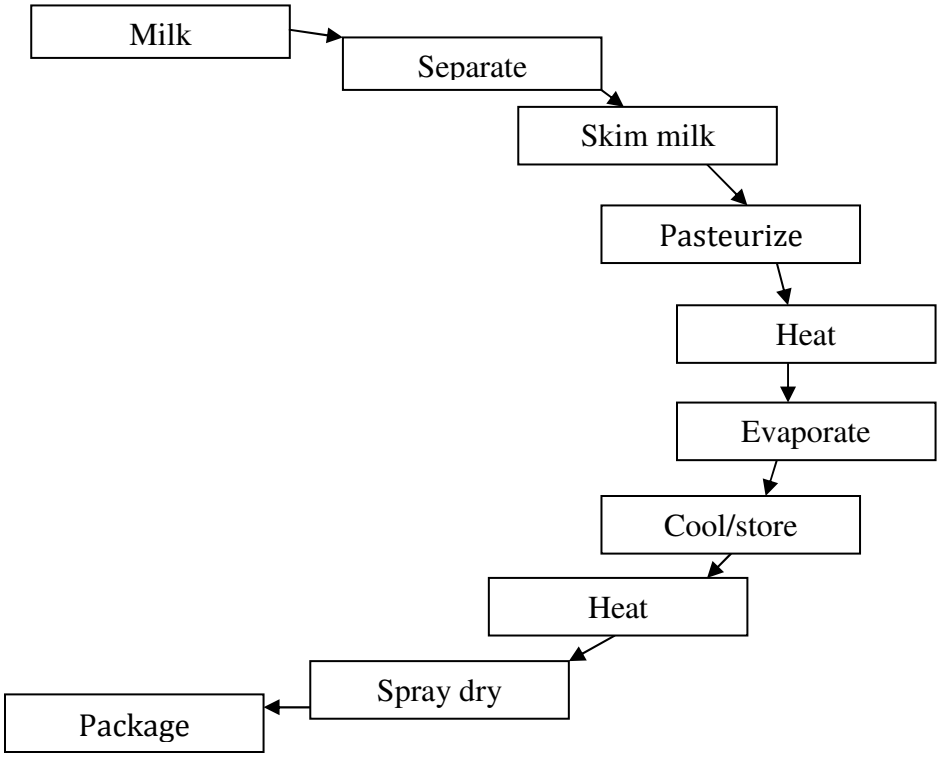

Figure 1: Manufacture of non fat dry milk

Nonfat dry milk and skimmed milk powder are classified for use as ingredients according to the heat treatment used in their manufacture. The heat treatment is measured based on undenaturated whey protein nitrogen index (WPN), applied to the milk during processing to milk powder (Cunniff, 1995). There are three main classifications: high-heat, medium-heat, and low-heat (21.CFR., 2006).

It is the basis of the following heat classifications:

Table 1: Whey Protein Nitrogen Index (WPNI) - Heat treatment

(American Dry Milk Institute, 1971)

Class	WPN
High heat powder	< 1.5
Medium heat powder	> 1.5 - < 6.0
Low heat powder	> 6.0

WPN is expressed as milligrams (mg) undenaturated whey protein nitrogen per gram of non-fat milk powder with a moisture content of 3.16%.

2.5.2. Definition of Dry Buttermilk/ Buttermilk Powder

Dry buttermilk product (made by the spray process or the atmospheric roller process) is the product resulting from drying liquid buttermilk that was derived from the churning of butter. The liquid buttermilk is pasteurized prior to condensing at a temperature of 161ºF for 15 seconds or its equivalent in bacterial destruction (Anonymous, 2009).

Buttermilk powder/ dry buttermilk typically contains 5% or less moisture (by weight) and 4.5% or more milkfat (by weight). Buttermilk powder/ dry buttermilk must have a protein content of not less than 30% (American Dairy Products Institute, 1990). It may not contain, or be derived from, nonfat dry milk/ skimmed milk powder, dry whey or products other than buttermilk, and contains no added preservatives, neutralizing agent or other chemicals. By removing moisture to the greatest extent possible, microbial growth is prevented (American Dairy Products Institute, 1990).

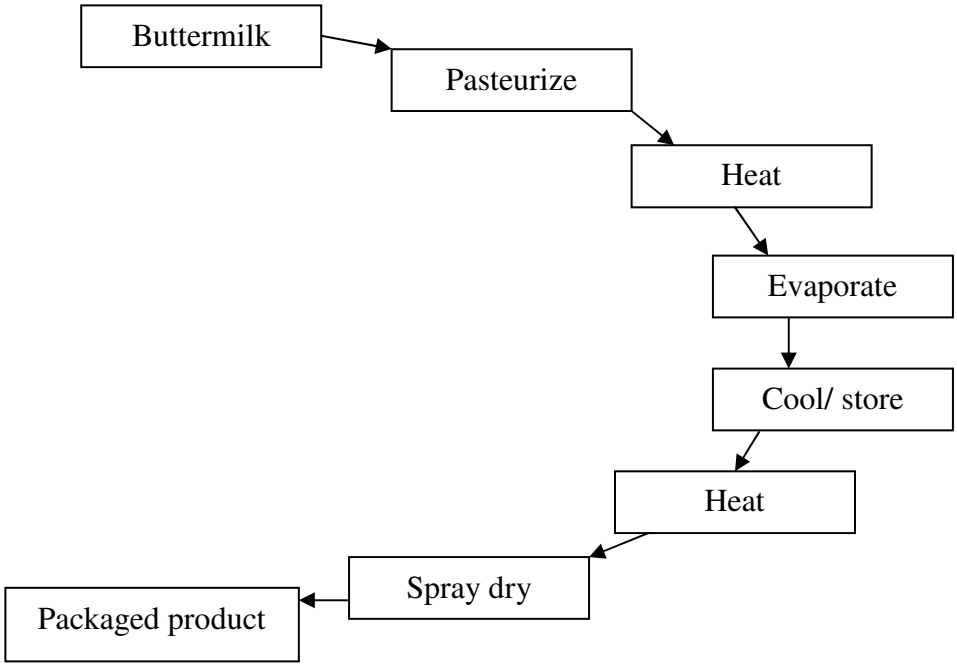

Figure 2: Manufacture of buttermilk powder (Anonymous, 2009)

2.5.1. Definition of Dry Whole Milk/ Whole Milk Powder

Dry whole milk/ whole milk powder is usually obtained by removing water from pasteurized, homogenized whole milk. It may also be obtained by blending fluid, condensed or skimmed milk powder with liquid or dry cream or with fluid, condensed or dry milk, provided the composition of the dry whole milk/ whole milk powder conforms to U.S. Federal Standards. Dry whole milk/whole milk powder must contain between 26% and 42% milkfat (by weight) on an “as is" basis. It must contain no more than 5.0% moisture (by weight) on a milk-solids-not-fat (MSNF) basis and not less than 34% milk protein on a milk solids-not-fat basis (Codex.Stan.207, 1999).

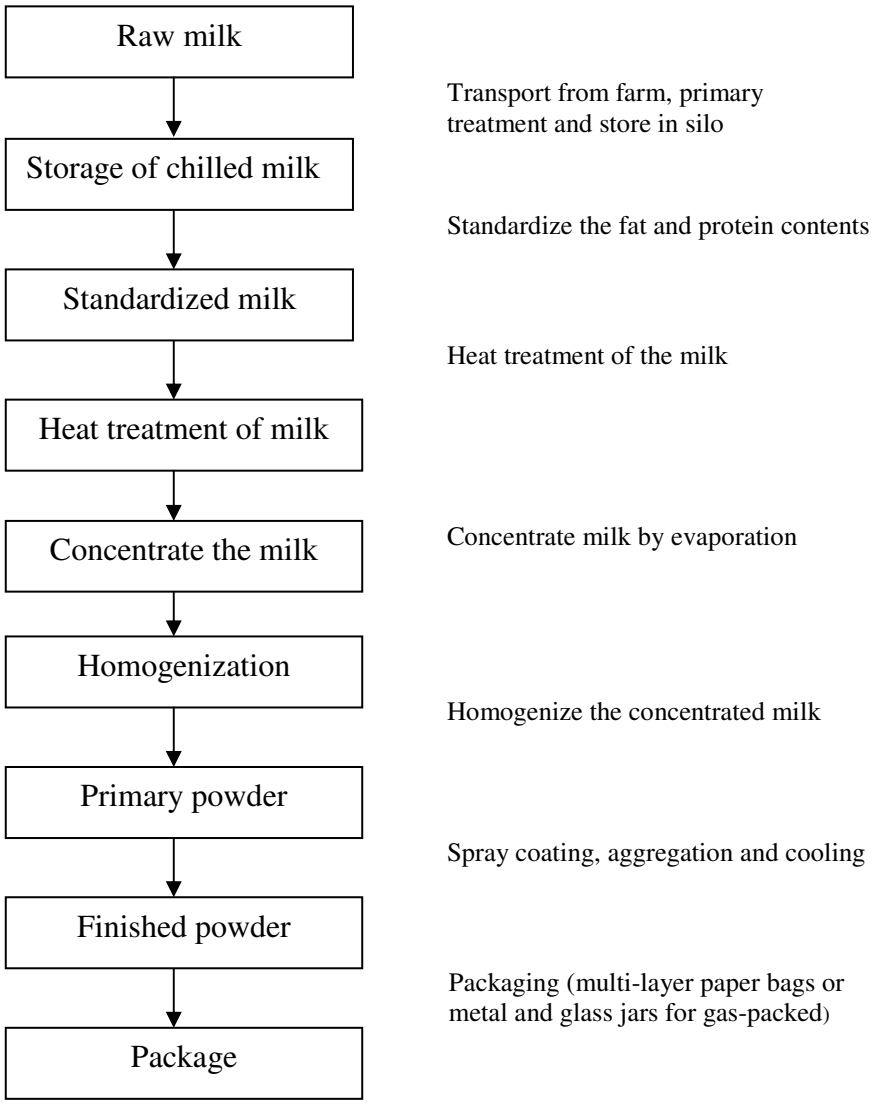

Figure 3: Manufacture of whole milk powder (Anonymous, 2009)

2.5.2. Definition of Whey Protein Concentrate

Whey is the liquid by-product from the cheese making process. Protein and fat in whey are recovered commercially by ultrafiltration (UF), because these molecules, by virtue of their size, are retained, while lactose and ash are able to pass through the membrane into the permeate. The retentate stream is then fed into spray dryers to produce powdered whey protein concentrate (WPC). Diafiltration (DF) water is usually added to the retentate in later stages of UF plants with large numbers of stages or loops, as they are

commonly known in the dairy industry. The addition of DF water increases the permeation of non-protein species across the membrane, thereby increasing the protein concentration of the total solids in the retentate stream. In fact, whey protein concentrate (WPC) is obtained by removing sufficient non-protein constituents, such as lipids, minerals and lactose, from pasteurized whey, for a dry product containing 34-80% protein (21.CFR.Ch184.1979., Spreer, 1998, Yee et al., 2007)

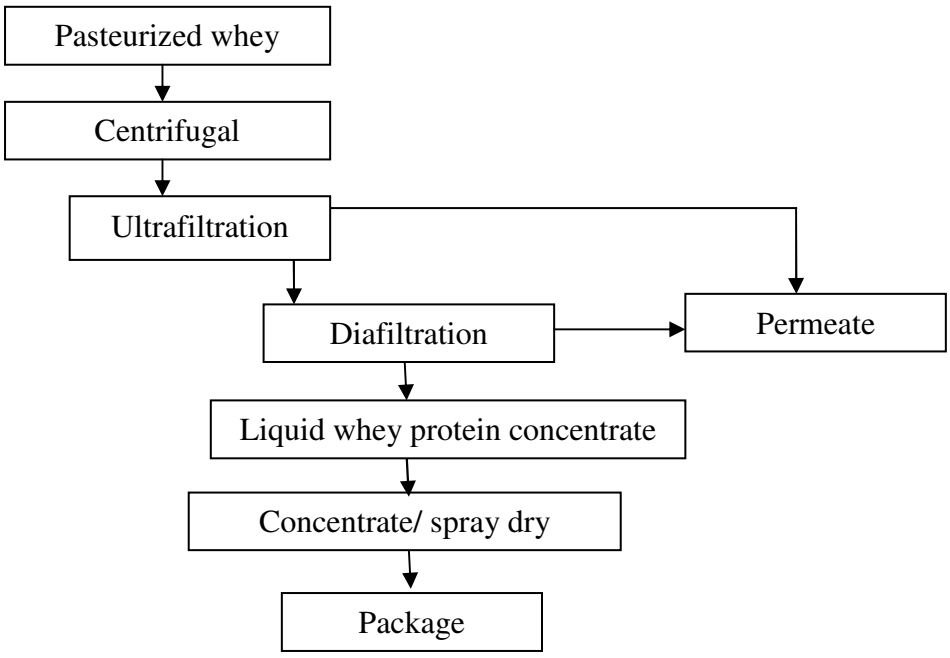

Figure 4: Manufacture of whey protein concentrate 80 (Anonymous, 2009)

2.5.3. Definition of Milk Protein Isolate

Milk protein concentrates (MPCs) typically range from 42 to 85% protein (dry basis). A milk protein concentrate having 90% protein is referred to as milk protein isolate (MPI). Milk protein isolate is produced by a combination of ion exchange and two pressure driven processes using semi-permeable membranes known as ultrafiltration (UF) and diafiltration (DF) and subsequent water removal processes. Water removal may

involve reverse osmosis, vacuum evaporation, and spray- drying. Lactose and minerals are removed until the desired protein content is reached (Varnam and Sutherland, 1994).

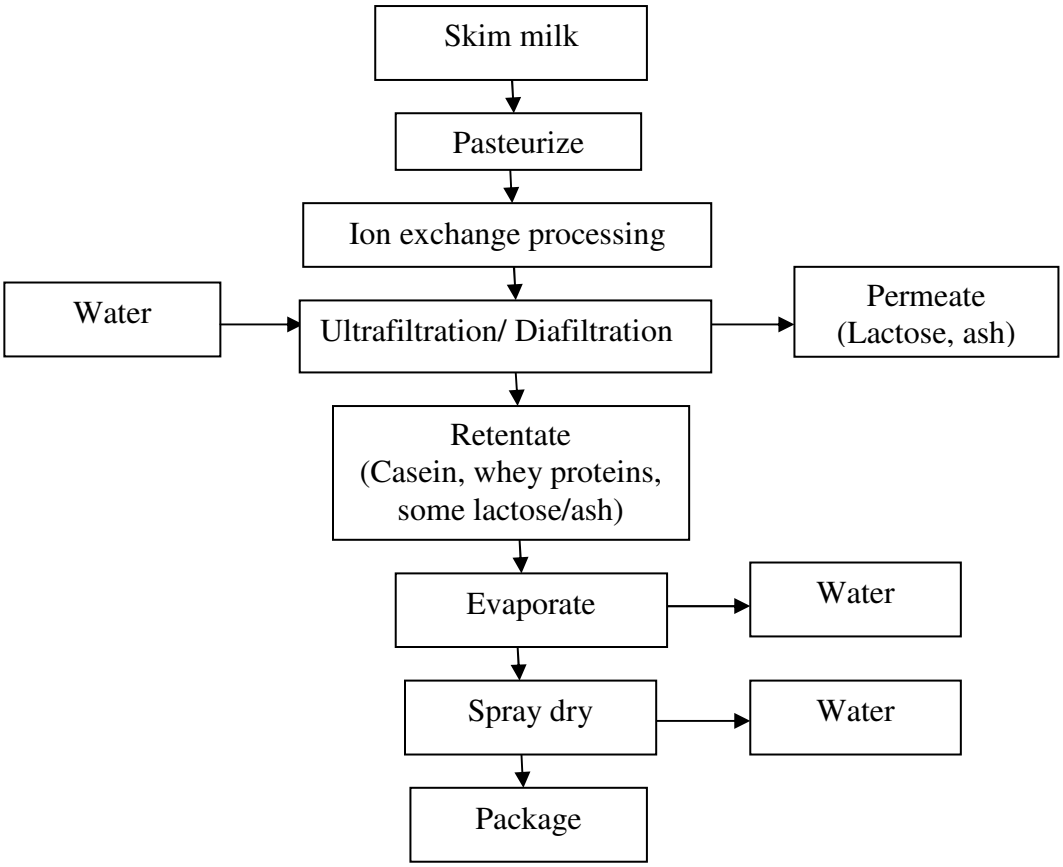

Figure 5: Manufacture of milk protein isolate (Anonymous, 2009)

2.5.4. Definition of Sweet Whey Powder

Sweet whey powder is obtained by drying fresh whey (derived during the manufacture of cheeses such as cheddar, mozzarella, monterey jack, and swiss) that has been pasteurized and to which nothing has been added as a preservative. Sweet whey powder contains all the constituents of fresh whey, except moisture and fat, in the same relative proportions as in the whey (21 CFR 184.1979).

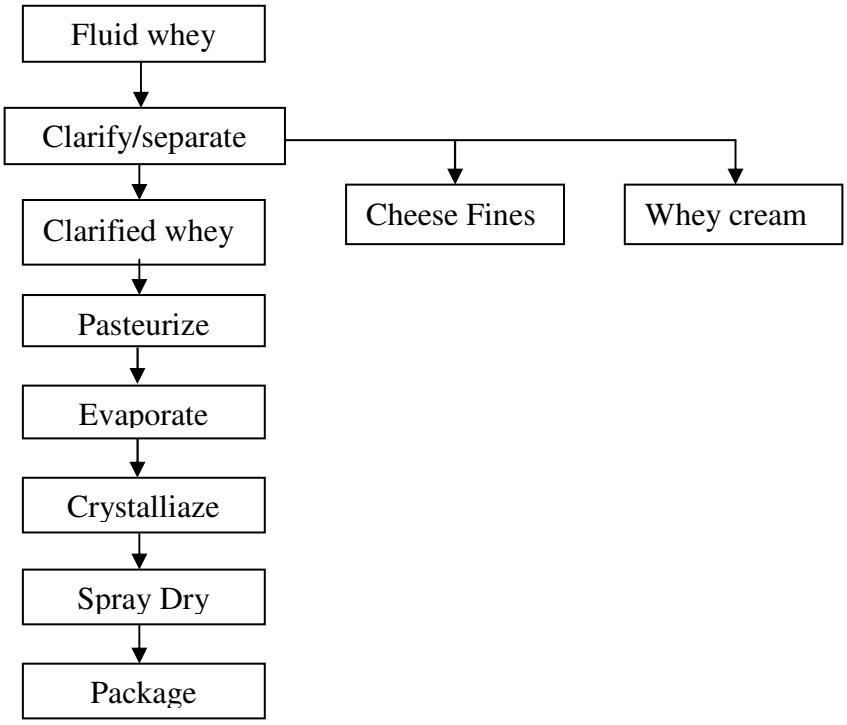

Figure 6: Manufacture of sweet whey powder (Anonymous, 2009)

2.6. Definition Of Shelf Life

The shelf-life of a product is defined as the finite length of time after production that a food will retain a required level of quality. In order to extend the shelf-life of a product, one must first assess the rate of change in food quality as it ages. Thus, it is important to identify the chemical and biological reactions influencing the quality and safety of the food. Once this is done, the reaction or reactions having the most critical impact on the rate of deterioration must be determined and dealt with (Taoukis et al., 1997). Usually, there is one limiting reaction that most affects the quality of the product and others are so excessive that their change in concentration with time is negligible.

The shelf-life of a product depends on four main considerations: formulation, processing, packaging, and storage conditions. Packaging is an integral part of the preservation system and functions as an interface between the food and the external

environment (Da Cruz et al., 2007, Robertson, 2006b, Sothornvit and Pitak, 2007). Packaging plays a fundamental role in maintaining the quality and therefore the shelf life of foods. For packaging and storage conditions, the keeping properties of the product are a function of its microenvironment. This includes temperature, relative humidity, gas composition, light, and pressure. Any or all of these components may be manipulated to lengthen shelf-life..

2.7. Expected Shelf Life of Dry Dairy Ingredients in the United States

2.7.1. Non Fat Dry Milk (NFDM)

The shelf life of non-instant NFDM is expected to be 12-18 months, if shipped and stored in a cool, dry environment at temperatures less than 27°C and relative humidity less than 65%. Under ideal conditions, non-instant nonfat dry milk powder can retain its physical and functional properties for at least two years; however, quality will be impaired if temperatures and humidity are too high and storage is extended (Farkye et al., 2001).

Driscoll et al. (1985) investigated the sensory quality of instant and regular skim milk powders after 4 years storage in cans and polybags at 10, 21 and 32◦C and in atmospheres of normal air, air modified with 100% CO_2 or 100% N_2. They found that powder stored under air had a much lower sensory quality than those stored under either N_2 or CO_2 at the same temperature.

2.7.2. Buttermilk Powder (BMP)

The shelf life of BMP is expected to be 6 to 9 months, if shipped and stored in a cool, dry environment at temperatures less than 27ºC and relative humidity less than

65%. Because Buttermilk contains a greater residual fat content than skim milk and a increased level of phospholipids with a higher fraction of multiple unsaturated fatty acids, buttermilk powder is prone to oxidation and has only a limited shelf life. Flavor quality will be impaired if the product is stored at too high a temperature for too long (USDEC, 2010a).

2.7.3. Whole Milk Powder (WMP)

The shelf life of WMP is expected to be 6 to 9 months, if shipped and stored in a cool, dry environment at temperatures less than 27ºC and relative humidity less than 65%. Milkfat is susceptible to oxidative reactions that are accelerated by increased temperature. Flavor quality, in particular, is impaired if storage temperatures are too high and storage is extended. Whole milk powder can have a shelf-life of more than 12 months if packaged in cans under vacuum or with an inert gas, such as nitrogen (Tamime, 2009).

2.7.4. Whey Protein Concentrates (WPC)

The shelf life of WPC is expected to be typically 9 to 12 months, if shipped and stored in a cool, dry environment with temperatures of less than 27°C and relative humidity less than 65%. WPCs have low bulk density and a tendency to dust; they are also very hydrophilic and absorb water from atmosphere very easily, therefore selecting the right packaging conditions is important to retain the quality of WPCs during the storage (USDEC, 2010b).

2.7.5. Milk Protein Isolate (MPI)

Although many manufacturers of MPI claim a 12-18 month shelf life for the product, there is a lack of scientific literature on the shelf life of milk protein isolate.

2.7.6. Sweet Whey Powder (SWP)

Native SWP has a shelf life of greater than 2 years at any storage temperature less than 53 °C. However, intermediate acid SWP must be stored at a temperature of less than 38 °C to attain a 2 year or longer shelf life. However, decreasing the pH even further has a drastic impact: high acid SWP must be stored at less than 20°C to attain a minimum of a 2-year shelf life (Dattatreya et al., 2007).

2.8. Package Environments

The packaging has to perform its functions in three different environments: physical, ambient, and human environments (Lockhart, 1997). Failure to consider all three environments during package development will result in poorly designed packages, increased costs, consumer complaints, and even avoidance or rejection of the product by the consumer (Robertson, 2010).

2.8.1. Physical Environment

This is the environment in which physical damage can be caused to the product, including shocks from drops, falls, and bumps, damage from vibrations arising from transportation modes, including road, rail, sea, and air; and compression and crushing damage arising from stacking during transportation or storage in warehouses, retail outlets, and the home environment (Robertson, 2010).

2.8.2. Ambient Environment

This is the environment that surrounds the package. Damage to the product can be caused as a result of exposure to gases (particularly O_2), water and water vapor, light (particularly UV radiation), and the effects of heat and cold, as well as microorganisms

(bacteria, fungi, molds, yeasts, and viruses) and macroorganisms (rodents, insects, mites, and birds), which are ubiquitous in many warehouses and retail outlets. Contaminants in the ambient environment such as exhaust fumes from automobiles and dust and dirt can also find their way into the product unless the package acts as an effective barrier (Robertson, 2010)

Essentially, packaging systems for milk powder must protect the powder from exposure to moisture, O_2, and light and anticipate the likely external environmental factors, which include temperature, time, relative humidity, light, and physical hazards. Packaging techniques have been developed for dry milk powders to eliminate or reduce O_2, and hence reduce fat oxidation. These techniques include gas flushing and use of oxygen absorbers (Hotchkiss et al., 2006).

2.8.3. Human Environment

This is the environment in which the package is handled by people, and designing packages for this environment requires knowledge of the strengths and frailties of human vision, human strength and weakness, dexterity, memory, cognitive behavior, and so on (Yoxall et al., 2007). For example, a package must contain information required by law, such as nutritional content and net weight and to maximize its convenience or utility functions, it should be simple to hold, open, use, and (if appropriate) reclose by the consumer (Yoxall et al., 2007). A range of variables influence packaging developments. In designing a packaging system, trends, prerequisites, conditions, and developments in the external environment must be taken into consideration (Sonneveld, 2000).

In fact, packaging should be designed and developed not only to contain the food product but also to protect it and add value to it, as its design may directly affect the purchase decision of the consumer.

2.9. Current Packaging Methods of Dry Dairy Ingredients

Ideally, the type and construction of the package depends on the type of dairy powder (e.g., skimmed, whole, buttermilk, etc.), the "surface area to volume" ratio of the package, the desired shelf life, the ambient storage, the transport environment, and the anticipated market environment; however, currently dairy powders are packaged into either plastic-lined multi-wall bags (25 kg) or totes (600 kg). Bags generally consist of several layers to provide strength and the necessary barrier properties. Moreover, dairy powders are often packaged under modified atmosphere to protect the products from oxidation, maintain their flavor, and extend their keeping quality. Some retail dairy powders are currently packaged into either metal cans or multilayer bags (Robertson, 2006).

2.9.1. Metal Cans

Packaging milk powder in metal cans has been highly popular for a long time, particularly for retail packaging. Cans are commercially available with capacities of 400, 900, 1800, and 2500g. The main reason for using metal cans is their excellent physical strength, durability, absolute barrier properties to moisture, O_2, and light, absence of flavor or odor, and rigidity (Robertson, 2006b).

Because bare steel is susceptible to corrosion, it is commonly electrolytically coated with a very thin layer of tin; in addition, an organic lacquer is applied to further

protect the metal from corrosion and avoid metal-food contact (Robertson, 2006b). Among the organic polymeric coatings, epoxy-phenolic lacquers are often used on tinplate, although waterborne polymer coatings have been playing an increasingly important role as well (Manfredi et al., 2005).

A recent concern has been the presence of natural and synthetic chemicals in foods that exhibit estrogenic effects and act as endocrine disrupters. Powdered milk (including infant formulas) may have hormonally active contaminants, introduced in the manufacturing process and leached from containers (Casajuana and Lacorte, 2004).

Bisphenol A (BPA) has been found to be one of the more potent anthropogenic estrogen mimics (Kim et al., 2001). It is a monomer used to produce (among other things) epoxy resins that are widely used to coat the interior of cans, leading to potential human exposure. Kuo and Ding, (2004) detected BPA in powdered milk and infant formulas on the Japanese market at concentrations from 45 to 113 ng g^{-1}.

The milk powder steel can is commonly cylindrically shaped and may feature a reclosable (tight fit) lid. In the standard version, the can features a cylindrical body with "can ends" on both ends. The can body is welded longitudinally, and the can ends are seamed onto the can body. To obtain appropriate closure (i.e., to maintain the integrity of the pack) an elastomeric compound is included in the end seam. In cans with a reclosable lid, it is common to seal the underside of the can end with an aluminum foil laminate to ensure integrity during storage and distribution.

Milk powder has a long shelf life when packed in metal cans due to their excellent barrier properties. The exchange of moisture and O_2 and the influx of light are not possible. As dairy powders with a higher fat content are more susceptible to oxidation,

and most powders are susceptible to deteriorative effects such as lumping and caking from moisture ingress, with adequately constructed cans, a shelf life in excess of 5 years is realistic, particularly when the products have been gas-flushed with N_2 to minimize the amount of available O_2. However, national food safety authorities often adopt a conservative approach by reducing the nominated shelf life (Robertson, 2010).

Nonfat dry milk (NDM) and powdered whey beverages are available at retail level in the United States and many other countries packaged in no. 10 cans (157×178 mm with a capacity of 3108 mL) in a reduced-O_2 atmosphere to prolong shelf life (up to 54 months). Lloyd et al. (2004) found that in the 10 US brands tested, wide variation existed in: headspace O_2, can seam quality, sensory quality, and vitamin A (with 6 of 10 brands entirely lacking vitamin A). The a_w of the brands ranged from 0.14 to 0.28 (a typical range), corresponding to 3-5% moisture content.

2.9.2. Mutilayer Pouches

In recent years, aluminum foil/plastic film laminates have been introduced as a replacement for the tinplate can. The laminates can be formed, filled, gas-flushed, and sealed on a single machine from reel stock (Robertson, 2006a). Such flexible pouches or sachets are well positioned to exploit the opportunities from convenience food markets. Flexible packages reduce the volume of traditional packaging such as metal cans, reduce transport costs, reduce the cost of the packaging, and require less material, thus minimizing post consumer waste (Twede and Goddard, 1998). However, in many developing countries milk powder in metal cans is still the preferred packaging option for larger capacities because of recloseability and the fact that the empty can can be reused as a household utensil (Robertson, 2010).

Milk powder packaged in pouches is commercially available in a capacity range of 250-2500 g. In addition, sachets with smaller capacities are also available to provide convenient single-serve portions of up to 35 g. As with metal cans, milk powder packaged in multilayer pouches is predominantly destined for retail distribution. The single-serve sachets are mainly distributed in developing countries because of the need to provide an affordable but highly nutritious food product. This type of retail distribution usually entails exposure to high humidity, high temperature, high levels of light, and relatively long storage times (Uppu, 2002). Maintaining the quality of milk powder in such small sachets is a challenge, given the very high surface area to volume ratio.

A 2-year shelf life for milk powder in portion packs is normally required when distributing in the relatively complex environments of developing countries. In countries with more highly developed economies a maximum shelf life of up to 12 months is common.

Commonly, a laminated multilayer pouch for milk powder must comprise a barrier to water vapor, O_2 (especially for WMP), and light. Aluminum foil is capable of providing such a barrier provided the foil does not have pin holes in it. Aluminum foil built into a flexible material provides a close-to-absolute barrier. Building into a flexible material is essential because the foil does not have any mechanical strength by itself and therefore needs protection from mechanical damage. A sandwich construction with two plastic layers – one on the inside, such as low density polyethylene (LDPE), so that the pouch can be sealed and one on the outside, such as biaxially oriented polypropylene (BOPP) or poly (ethylene terephthalate) (PET), to provide mechanical protection and also carry information-is common practice (Uppu, 2002).

Alternatively, with pouches for which a shorter shelf life is acceptable, the alufoil layer may be replaced with a high-barrier plastic layer such as a copolymer of ethylene vinyl alcohol (EVOH) or polyvinylidene chloride (PVdC), possibly with the addition of a thin layer of metal or silica oxide (SiO_x) deposition to enhance its O_2 barrier characteristics (Lange and Wyser, 2003). However, the shelf life will likely be less than that of a pouch containing an Aluminum foil layer. A shelf life of up to 2 years is not feasible with portion pouches in a challenging distribution environment, such as exists in many developing countries, other than with the inclusion of an Aluminum foil layer. Sachets with larger capacity (in excess of 250 g) comprising a high-barrier plastic layer sandwiched between LDPE and BOPP or PET would be able to achieve a similar shelf life to an alufoil-sandwiched portion pack pouch (Robertson, 2006b).

Another material common in dairy powder packaging is called kraft paper, named after the process used to produce it from the pulp of softwood. Kraft is a strong and relatively coarse paper which usually comes in brown color. Its typical packaging applications are: paper grocery bags, envelopes, multiwall sacks, and dairy powder packaging.

There are two types of bag construction in dairy powders packaging; one in which the polyethylene (PE) liner is glued onto the innermost paper ply. The second type of construction is the cap sac bag in which the inner liner is separate from the paper bag. This bag style is also called bag-in-bag. The inner LDPE layer varies in thickness from 1 to 4 mil (1 mil = 0.025 mm). The common bag for dairy powders packaging consists of 3-ply Kraft paper and an inner of 3-mil LDPE liner. Plastic bags are also used for milk powder packaging which consist of two layers; an inner 3-mil LDPE liner and an outer

shell of 5-mil polypropylene (PP). PP is a hydrocarbon polymer polymerized from propylene gas and has good moisture barrier properties (Sokora, 1999).

2.10. Deteriorative Reactions and Indicators of Failure

2.10.1. Cohesion/ Flowability

Powder deposition on processing equipment is a problem in the dairy industry and results in economic disadvantages. Cohesion increases with a reduction in particle size. More surface area is available for cohesive forces, in particular, and frictional forces to resist flow (Fitzpatrick et al., 2004). Abbott (1990) reported that wall deposition in spray dryers may pose a potential fire risk, compromise hygiene requirements, and reduce product quality as well as yield. Spray dryer hazards include ignition of explosible dust clouds, dust deposits, bulk powder deposits and flammable vapor. Kieviet (1997) noted that wall deposition affected the residence time distribution of particles, and particularly that an important factor in determining residence times with high wall deposition rates was the time taken by particles to slide down the conical wall of a spray dryer. Sticking of particles to the walls and to each other, and sliding of wall deposits, are therefore important issues (Kota et al,. 2007).

Fat also plays an important part in the observed trend toward higher cohesion with increasing temperature. Melting of fat is likely to cause the major increase in cohesion, but there are several possible mechanisms (Rennie et al., 1999). The liquid fat may have formed bridges between the particles, which increase the bonding stretch. Alternatively, fat liquefaction could have softened the powder, resulting in deformation of powder particles, which would have increased the contact area between the particles, thus

enhancing already present attractive forces (Rennie et al., 1999). During processing, the behavior of powders is strongly influenced by particle properties as well as the design and operating conditions of the equipment. The flowability of powders in such equipment is an important issue as it can strongly influence the efficiency and reliable operation of these processes (Moreno-Atanasio et al., 2005). Intuitively, one would expect particle shape to affect flowaility, as shape will influence the surface contacts between particles; however, there is not much reported on the influence of shape on powder flowability (Fitzpatrick et al., 2004).

2.10.1.1. Interparticulate cohesive forces

The various interparticulate cohesive forces (Figure 7) involved in stickiness are (Rahman, 2003):

- Liquid bridges
- Solid bridges
- van der Waals forces
- Electrostatic forces
- Mechanical interlocking

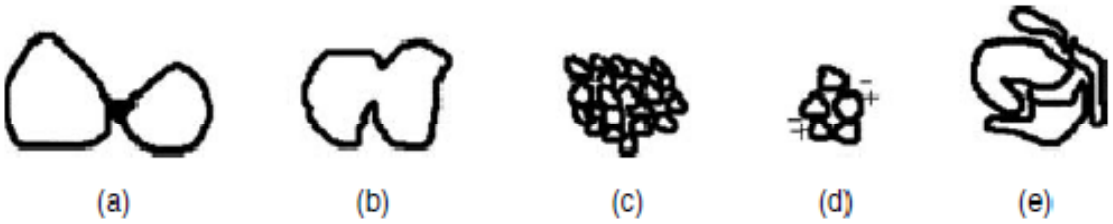

Figure 7: Schematic diagram of interparticulate cohesive forces. (a) Liquid bridges (b) solid bridges (c) van der Waals forces (d) electrostatic forces and (e) mechanical interlocking (Rahman, 2003).

Liquid Bridges

Liquid bridges are produced due to melting, wetting, and dissolution of the external surface of the particles or release of mobile liquid components from the interior of the particles (Figure 7a). This type of cohesion is mainly dominated by the surface tension and capillary properties (Rahman, 2003). For example, during rewetting of the particles in an agglomeration process, there is a flow of liquid between two adjacent particles. On removal of solvent (such as water), the mobile liquid bridges turn into solid bridges. However, this type of solid bond can be fragile due to the narrowness of the bridge. The presence of low-melting point components (such as oil) will also result in

liquid bridges (Rahman, 2003). This type of nonaqueous bonds is weak due to the noncompatibility with other solids present in the powder. The powder flow is adversely affected by the presence of liquid bridges. Many high fat powders do not flow well for this reason. Solidification of these liquid bridges due to temperature fluctuation makes the bond stronger (Rahman, 2003).

Solid Bridges

Solid bridges between the particles are formed by melting and solidifying (sintering), and crystallization of dissolved solids (Figure 7b). The interparticulate contact area is large and the strength of the agglomerate is high. Lowering of the temperature of the powder converts the liquid bridges into solid bridges. In some powders such as milk powders or high-sugar powders, this type of bond is so strong that it will need a hammer to break the lump (Rahman, 2003).

van der Waals Forces

All molecules possess weak attraction forces at very close distances. This is due to the electrostatic attraction of the nuclei of one molecule to the electrons of the other. This may result in polarization of the molecules at the surface. Fine powder particles (<1 μm in size), which have very small interparticulate space, tend to stick to each other due to this force (Figure 7c) (Rahman, 2003). The oscillation of the molecules and vibration of the bonds may also cause such attractions due to facilitation of the alignment of the positive and negative forces. When the force of gravity (e.g., larger particles) is larger than the van der Waals forces, the particles do not show such cohesive behavior. Generally, high-molecular-weight materials having more electrons tend to be more cohesive. The fundamental mechanism of the van der Waals forces is electrostatic in

nature (Feng and Hays, 2003). This type of stickiness will be common in submicron or nanoparticles. Deposition of fine powders into the dryer wall, equipment surfaces, and room walls is the result of van der Waals forces. This force is relatively weaker and therefore can be broken easily. However, owing to close proximity and minimum interparticulate space, this force accelerates other type of caking.

Electrostatic Forces

There is normally confusion over electrostatic forces and van der Waals forces. The van der Waals is a short-range force, whereas the electrostatic is a long-range force. The fine particles can have some excess electrons due to friction. If these excess electrons are not dissipated (due to low conductivity), the electron-rich particles can realign themselves with electron-poor (oppositely charged) particles to balance the charge (Figure 7d). This results in cohesion or adhesion of particles. In fact, van der Waals forces and electrostatic forces act in combination in the case of cohesion/adhesion of fine powders (Feng and Hays, 2003).

Mechanical Interlocking

This occurs due to the irregular and uneven shape and size of the particles (Figure 7e). The fibrous, bulky, and flaky particles will interlock with each other or "bird nest" (Barbosa et al., 2005). Under compaction or vibration, particles will reposition and become more entangled. On heating, wetting, and drying, these physical bonds can become very strong.

The energy of interactions between particles depends on the type of material, moisture content, size, and shape of the particles and external electrical field. In general, the solid bridges are stronger than the liquid bridges. The van der Waals forces are the

weakest one and their dominance is high when particles are very small and gravitational effect becomes nominal. The interlocking energy depends on the surface roughness of the particles and the amount of distortion and packing (Rahman, 2003).

2.10.2. Caking

Several properties of powders with amorphous lactose can be related to its glass transition tempreture T_g. These properties include surface stickiness and caking, time-dependent lactose crystallization and release of encapsulated lipids, and increasing rates of nonenzymic browning and lipid oxidation. When an amorphous component is given suitable conditions of temperature and water content, powder can mobilize as a high-viscosity flow, which can make it sticky and lead to caking (Fitzpatrick et al., 2007). The changes in mechanical properties and diffusion are responsible for stickiness, caking and lactose crystallization (Aguilera et al., 1995). Caking is a deleterious phenomenon by which a low-moisture, free-flowing powder is first transformed into lumps, then into an agglomerated solid, and ultimately into a sticky material, resulting in loss of functionality and lowered quality (Aguilera et al., 1995). Amorphous lactose is generally present in high-fat powders and can contribute to flowability problems; however, these problems also arise under conditions [a_w (water activity) and powder temperature] where the amorphous lactose is stable (Foster et al., 2005a). This indicates that although packaging and storage conditions (specifically, water activity and tempreture) may influence caking in milk powders, milk fat also contributes to caking (McKenna, 1997, Peleg, 1977). The changes in the reaction rates are more complex and affected by other factors, including pH, heterogeneities in water distribution, and miscibility of proteins and carbohydrates (Roos, 2002). The next sections discuss the factors responsible for caking explicitly.

2.10.2.1. Crystallization, a Major Reason Responsible for Caking

The crystallization behavior of amorphous lactose in dairy powders is a temperature dependent phenomenon. Berlin et al. (1970) observed that the relative humidity at which the break in sorption isotherms appeared was dependent on temperature, which was confirmed by Warburton and Pixton (1978). Dairy powders contain amorphous lactose, which is unstable and will try to crystallize when given suitable conditions of temperature and moisture content that allow the molecules to mobilize and initiate crystallization (Fitzpatrick et al., 2006). The glass transition temperature is very important, because the amorphous component can start to mobilize above this temperature. Higher temperatures produce greater mobility, which leads to greater rates of crystallization and caking (Fitzpatrick et al., 2006). Dairy powders are hygroscopic powders which readily sorb moisture from their surrounding environment and this reduces the glass transition temperature making them more susceptible to caking (Fitzpatrick et al., 2006). Moisture sorption may result in the formation of liquid bridges which can lead to the powder becoming much more cohesive and eventually caking. This can cause problems in powder handling (Fitzpatrick et al., 2006).

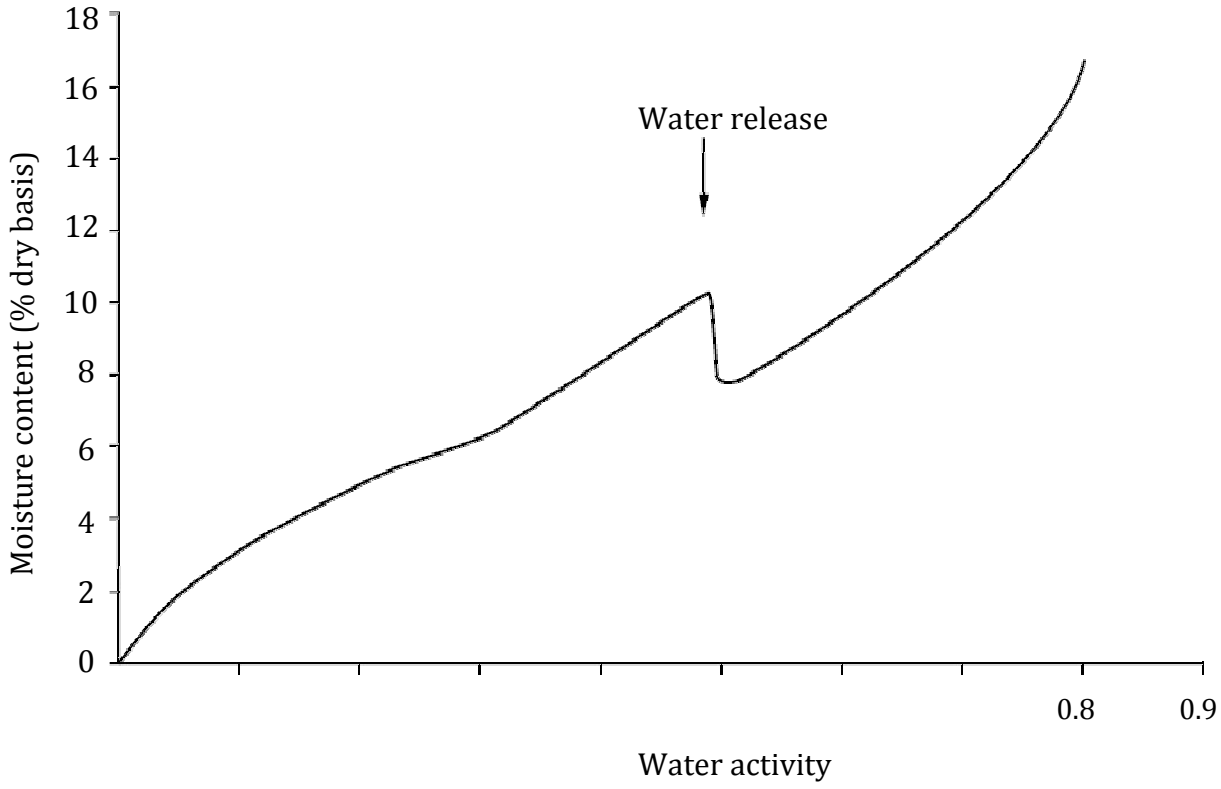

Figure 8: Generalized moisture sorption isotherm for milk powders showing a break at $a_w = 0.5$, due to lactose crystallization (Thomas et al., 2004)

Although lactose is highly hygroscopic, crystallization does not occur if $a_w < 0.34$, the moisture content < 8.4%, and storage temperature < 20°C (Vernam and Sutherland, 1996). A generalized moisture sorption isotherm (MSI) for milk powders was shown in Figure 8 with a break at $a_w = 0.5$, where water is released due to lactose crystallizing (Thomas et al., 2004a). With the relatively high lactose contents in whole milk powder (WMP), non fat dry milk (NFDM), and sweet whey powder (SWP), (30, 50 and 70%, respectively), the powders may be prone to caking with an increase in free moisture due to lactose crystallization. Difficulties in dispersing the powders in water (i.e., diminishing the solubility) may be the result.

Figure 9 shows scanning electron microscopic pictures of whole milk powder containing major amounts of amorphous substances. Some inter-particle bridges are highlighted by white circles.

Figure 9: Scanning electron microscopic picture of spray-dried whole milk powder containing crystalline lactose (Aguilera et al., 2011)

2.10.2.2. Other Factors Responsible for Caking

Presence of liquid component

Some of the components in dairy powders can be in liquid state such as fat. In powder, such liquid state should be in discontinuous phase, so to be encapsulated by the solid continuous phase. However, leaching or breaking the structure can cause release and coalescence of such liquid at the surface of particles. This results in particles sticking together. This is manifested by decreased flowability and sluggish behavior of powder (Rahman, 2003). Free fat/oil in the powders causes caking, but the cakes are not as strongly held together as in the case of caking as a result of other factors such as lactose crystallization in milk powder during storage. The increased temperature can also result in melting of the solid phase (fat to oil) or state change (glassy to rubbery), consequently resulting in caking of powder (Rahman, 2003).

Moisture absorption

Water absorption by the powder can cause dissolution of outer surface of the particles or condensation of the capillary moisture in amorphous material. This eventually creates a liquid bridge. Upon dehydration, these liquid bridges are converted into strong solid bridges. The distance between the particles and hygroscopic property of the particle components will influence the rate of caking. The higher the compactness of the particles and the finer the particles, the faster will be the caking (Rahman, 2003).

Consolidation

Consolidation or compression of powders decreases the distance between the particles, consequently van der Waals and other forces become predominant. This results in caking. This is more important if the powders are fine or brittle or break due to the compression force. The filling of the voids during compression, absorption of moisture, leakage of liquid fraction, particle shape and size, and increased bulk density will contribute to caking during compression (Fitzpatrick, 2005). Tuohy (1989) found considerable differences between the packed bulk density of regular SMP (0.85 g/cm3), WMP (0.68 g/cm3) and fat-filled milk powder (0.47 g/cm3). The packed bulk density of fat-filled milk powder was about 50% of that of SMP, suggesting that fat also influences the consolidation of milk powder. Caking caused by consolidation may be a concern in regard to possible changes in density, flowability and particle size of vacuum packaged dairy powders and it is well worth further investigation.

2.10.3. Maillard Reactions

Maillard reactions are an important class of deteriorative reactions in milk products. This type of chemical reaction is initiated by condensation of lactose with the

free amino group of lysine in milk proteins (Thomsen et al., 2005). In milk products such Maillard reactions are induced by heating during processing and long term storage at moderate to high temperature (O'Brien and Morrisey, 1989). Crystalline forms of lactose depend on the preservation time and many other conditions, such as humidity, storage temperature, and manufacturing process. The crystalline state is thermodynamically favored as it has a lower free energy due to structured arrangement of the molecules. During crystallization, the amorphous lactose will initially absorb moisture from the surroundings due to its hygroscopic nature, and subsequently release moisture as it crystallizes, as shown in Figure 8. The crystallization kinetics can be determined from the mass change of the powder (Ibach and Kind, 2007). Lactose crystallization modifies the microstructure and chemical composition of the surface of powder particles (Thomas et al., 2004a).

Dairy powders are sensitive to Maillard reaction as they contain high concentration of lactose and proteins with high lysine level (Palombo et al., 1984). In addition, relatively high temperature and water content during processing and prolonged storage, are the major factors involved in the high susceptibility of dehydrated dairy products, as they are favorable conditions for the Maillard reaction (Labuza, 1972). Maillard reaction in dairy powders is also important as it also causes off flavors. These off-flavors are generally characterized as "caramelized" or "toasted" (Farkye et al., 2001). At high storage temperatures, these types of flavors have been described in dried milk products. The off-flavors in dairy powders can influence their market acceptance depending on the applications. For instance, a slight caramelized taste is objectionable in products such as yogurt, ice cream and similar products; however, it is not a problem in

other food applications such as in baked goods and some dry blends. One of the most obvious negative consequences of the Maillard reaction in food is the loss of nutritive value of proteins involved, with a loss of quality and a possible decrease of food safety. It has been shown that the Maillard reaction leads to the loss of nutritional value in milk powder (Erbersdobler & Somoza., 2007).

2.10.4. Lipid Oxidation

Lipid oxidation is one of the most basic chemical reactions that occur in milk and milk products, generally resulting in deterioration in sensory and nutritional quality. Lipid oxidation, by definition, requires the presence of oxygen; however, the minimum residual oxygen concentration may vary between different dairy products. Products with a large surface area such as dairy powders should theoretically be more predisposed to oxygen exposure and hence lipid oxidation.

Many reviews of the chemistry of lipid oxidation have been published (Belitz and Grosch, 1999, Chan, 1987, Frankel, 1980, Frankel, 1982, 1985, 1988, 1991, 1998, Gardner, 1989, Grosch, 1987, Kochhar, 1996, Kolakowska, 2003, Labuza, 1971, Min and Lee, 1996, Richardson and Korycka-Dahl, 1983, Schaich, 1980, St. Angelo, 1996). Lipid oxidation is essentially a free-radical chain reaction involving initiation, propagation and termination stages. The reaction of unsaturated lipids with molecular O_2 results in the formation of hydroperoxides, which then break down to off-flavor compounds (Liang, 1999). Many factors are responsible for degradation of lipids due to oxidation, and one of the major causes of this defect has been identified as the oxidation of unsaturated lipids (Cadwallader and Howard, 1998). Lipid peroxidation off-flavors are caused by the formation of secondary reaction products (alkanes, alkenes, aldehydes, and ketones)

(Romeu-Nadal et al., 2007). These compounds impart off-flavors and loss of nutrients to milk powders and thus limit their shelf life stability (Fenaille et al., 2003). Lipid oxidation in WMPs is a major cause of deterioration during processing and storage (McCluskey et al., 1997).

Packaging and storage conditions (oxygen, light exposure, storage temperature, moisture) as well as compositional properties (water content and percentage of unsaturated fatty acids), and process parameters are the most important factors that affect oxidation. Inhibiting the progress of lipid oxidation in milk and milk products, including dairy powders, is a key factor in maintaining quality and extending shelf-life (O'Connor and O'Brien, 1995). The next section would discuss the main factors influencing oxidation of dairy powders.

2.10.4.1. Mechanism of Lipid Autoxidation

The hydroperoxide theory of the oxidation of unsaturated lipids is universally accepted. The fundamental principles were elucidated by the work of Farmer et al. (1942), Bolland and Gee (1946) and Bateman et al. (1953). The initial step in the autoxidation of unsaturated fatty acids is the formation of free radicals. The formation of the initial free radical to start the oxidation process may be due to factors such as irradiation, metal complexes, enzymes or active oxygen species (O'Connor et al., 1995). In the case of monounsaturated and nonconjugated polyunsaturated fatty acids in milk lipids, the reaction is usually initiated by removal of a hydrogen from the methylene group adjacent to the double bond (O'Connor et al., 1995).

Food lipids possess an inherent stability to oxidation, which is influenced by the presence of antioxidants and pro-oxidants (O'Connor et al., 1995). After a period of

relative stability (induction period), lipid oxidation becomes autocatalytic and rancidity develops. Thus, the typical time-course of autoxidation, as measured by the concentration of hydroperoxides, consists of a lag phase (induction) followed by the rapid accumulation of hydroperoxides, which reaches a maximum and then decreases as hydroperoxide decomposition reactions become more important. The longer the induction period, the more stable the food to oxidation (Lundberg, 1962).

2.10.4.2. Factors influencing oxidative stability of dairy powders

Water Activity

The shelf life of high fat milk powders, such as whole milk powder, depends on the preheat treatment of the milk, the water activity (a_w) of the product, and the storage temperature. One of the factors influencing the rate of autoxidation in milk powder, although less investigated, is a_w.

The relationship between water content and water activity is complex. An increase in a_w is almost always accompanied by an increase in the water content, but in a nonlinear trend (Bell et al., 2000).

This relationship between water activity and moisture content at a given temperature is called the moisture sorption isotherm (Bell et al., 2000). Moisture sorption isotherms are sigmoidal in shape for most foods, and a moisture sorption isotherm prepared by adsorption (starting from the dry state) will not necessarily be the same as an isotherm prepared by desorption (starting from the wet state). This phenomenon of different a_w vs moisture values by the two methods is called moisture sorption hysteresis and is exhibited by many foods (Figure 10). Hysteresis represents the difference in a_w between the absorption and desorption isotherms (Bell et al., 2000).

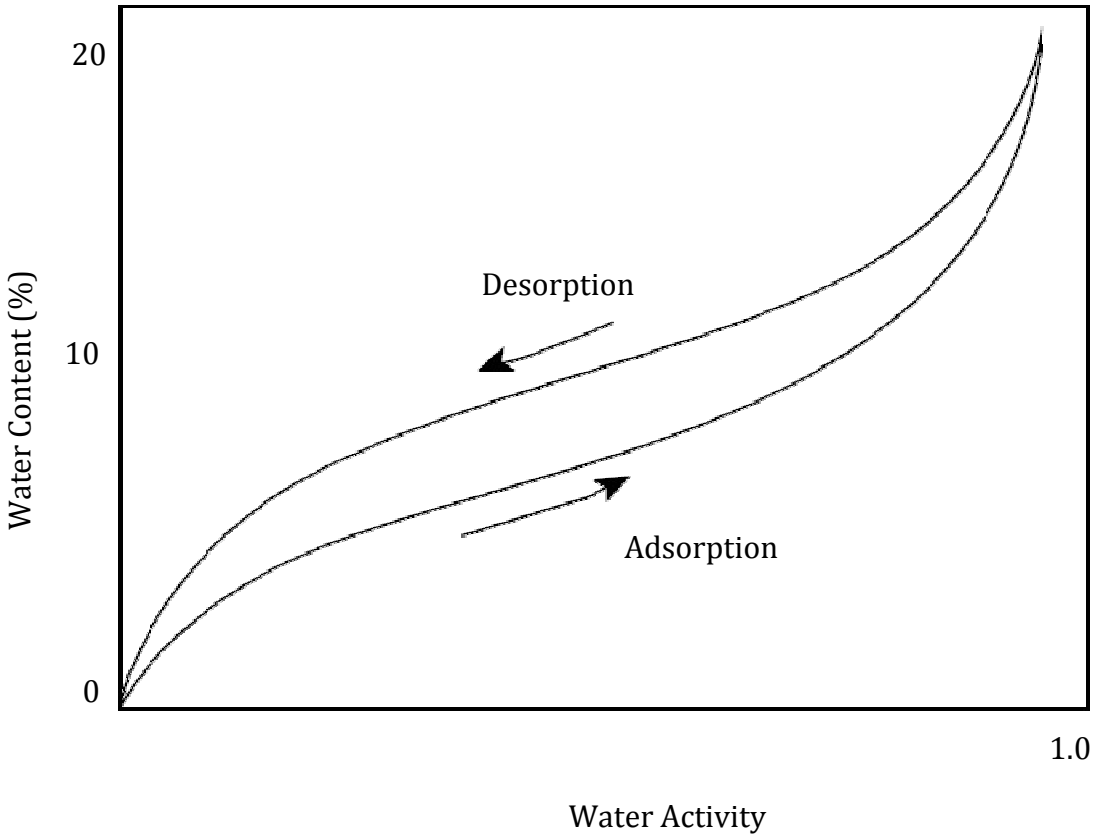

Figure 10: Water sorption hysteresis

The maximum shelf-life of bulk packaged WMP containing 3% moisture is about 6 months at 30ºC (Kjargaard Jensen, 1988). The oxidation of WMP, as measured by peroxide value, is dependent on the moisture content of the powder. van Mil and Jans (1991) reported that under similar storage conditions, the peroxide value of WMP increases more rapidly for powder containing 3% moisture than in powder containing 2.4% moisture. The water activity (a_w) range for WMP is usually 0.13–0.20, with a typical value from 0.16 to 0.18 (Wewala, 1990). Loncin et al. (1968), Roos (2002) and Stapelfeldt et al. (1997) found that autoxidation in an unspecified milk powder, as

measured by peroxide values, was stimulated by an a_w below 0.11 and unaffected by (a_w)s between 0.11 and 0.75

Stapelfeldt et al. (1997) found that the quality of WMP is maintained best at a_w between 0.11 and 0.23, whereas the quality of the powder decreases when stored at a_w of 0.31 at 458C. However, the critical a_w for improved oxidative stability of WMP stored at 40ºC for one year is 0.21–0.24 at a moisture level of 3.4% (Wewala, 1990).

Temperature

The preheat treatment of milk prior to the manufacture of milk powder is the major factor controlling the oxidative stability of the product, as heat treatment at high temperatures, apart from increasing the microbial safety, delays the onset of oxidized flavor, which is the limiting factor for the storage of milk powder (Baldwin et al., 1991). Stapelfeldt et al. (1997), Thomsen et al. (2005), and Augustin et al. (2006) found that long-term stability of milk would be influenced negatively by a low preheat intensity, a high storage temperature, and a high a_w during storage. Although the effect of preheat treatment and storage was in qualitative agreement with earlier findings, the effect of a_w should be noted, especially as these findings were further substantiated by the techniques used to follow different stages of oxidation in the main experiment. There has been increasing interest in the supplementation of milk powder formulas with long-chain polyunsaturated fatty acids (LC-PUFAs) especially with arachidonic acid (C20: 4n-6, AA) and docosahexanoic acid (C22: 6n-3, DHA). High temperatures and the presence of O_2 lead to increased oxidation of PUFAs (Romeu-Nadal et al., 2007).

Oxygen

As O_2 is consumed during oxidation, the O_2 content will also influence lipid oxidation. In addition, the O_2 concentration in the headspace and the product is important, as this can influence the oxidation rate. Oxygen concentration could also influence the oxidation pathways and lead to different oxidation products (Grosch et al., 1981). It has been shown by numerous authors that if O_2 in milk powder or infant formula packages is replaced by N_2 and CO_2, the oxidation is not detectable and the peroxide value does not increase (Van Mil and Jans, 1991). Oxidation increases during storage; for example, WMP has a maximum shelf life of 6 months at room temperature (Anon, 1989). However, it was found that WMP could have a shelf life in excess of 12 months if it was packed in cans under vacuum or an inert gas such as N_2 to inhibit the development of off-flavors (Kieseker and Aitken, 1993).

The amount of O_2 needed to cause unacceptable oxidative changes is usually very small (Labuza, 1971). There is little detailed knowledge about what levels are acceptable for specific food products and how the storage stability is related to the amount of O_2 available for oxidation, especially at very low O_2 levels, that is, below 1 mL L^{-1}. Andersson and Lingnert (1997) reported on the influence of O_2 levels down to 0.6 mL L^{-1} on the oxidation of cream powder. An increased temperature also increases the effect of O_2 concentration. At high partial pressures of O_2, the oxidation rate should, theoretically, be independent of O_2 concentration and be directly dependent on substrate concentration (Labuza, 1971).

Many researchers have reported significant improvements in the sensory quality and shelf life of milk powders stored in the absence of oxygen; although most of these studies involved whole milk powder (Andersson and Lingnert, 1998, Chan et al., 1993,

Coulter, 1947, Min and Lindamood, 1989, Tuohy, 1984, Warmbier and Wolf, 1976). It is therefore not surprising to expect an extended shelf life for vacuum packaged milk powder. This is particularly true about dairy powders, the shelf life of which is governed to a large extent by the rate of oxidation of the unsaturated fats and the consequent development of objectionable flavors, in products such as whole milk powder.

Although there has been a general consensus of opinion among investigators that reduced levels of oxygen in the package retard the development of oxidized flavor, especially in whole milk powders, no agreement exists as to the minimum levels needed during storage. Lea et al. (1960) concluded that the development of oxidized flavors in spray-dried whole milk could be controlled by reducing the oxygen content in the free space of the container to 1 to 3%. Coulter (1947) and Coulter et al. (1948) concluded that less than 1% oxygen in the packaging gas was necessary to prevent oxidation of the whole milk powder. Shaffer, (1945) reported 3% as the upper limit of in package oxygen content for extended storage life at room temperature.

Lloyd et al. (2009) evaluated the influence of packaging atmosphere, storage temperature and storage time on the shelf life of WMP, using sensory and instrumental techniques. They showed that:

(1) Air-packaged WMP had higher peroxide values, lipid oxidation volatiles, and off flavors (grassy and painty) than nitrogen-flushed WMP.

(2) The storage temperature did not affect levels of straight chain lipid oxidation volatiles, as 23°C storage resulted in higher cooked and milkfat flavors and lower levels of grassy flavor compared to 2°C storage.

(3) Consumer acceptance was negatively correlated with lipid oxidation volatiles and painty flavor. Finally, they concluded nitrogen flushing prevents the development of painty flavor in WMP stored up to 1 yr at either temperature (23 or 2°C).

Packaging is used to exclude, control, or contain O_2 at the level most suited for a particular food product. T o prevent the oxidation of milk powders, the packaging must provide a high-level O_2 barrier and be able to retain that barrier during the anticipated shelf life. Oxygen transmission rate (OTR), is a measure of the passage of oxygen through a packaging material and it is defined as the steady state rate at which oxygen gas permeates through a film at specified conditions of temperature and relative humidity. Values are expressed in cc/100 in^2/24 hr in US standard units and cc/m^2/24 hr in metric (or SI) units. Standard test conditions are 73°F (23°C) and 0% RH (ASTM-D3985).

There are two methods for reducing product exposure to oxygen via flexible packaging: modified atmosphere packaging and vacuum packaging.

Modified Atmosphere Packaging (MAP) is a process for replacing the air in the headspace of a package with another gas before the final seal is made. This is also called gas flushing. The most common replacement gases are nitrogen or nitrogen/carbon dioxide mixtures (Hui, 2005).

Vacuum Packaging is a technique for packaging food products. It involves the extraction of the air contained in the package and then hermetically sealing it. In this way oxygen and all chemical and biological contaminants such as pollution substances, bacteria and mold that are normally present in the air, are eliminated from being in contact with the product. Different degrees of vacuum can be used as a function of

packaging machine regulation and product characteristics, to arrive at an almost complete extraction of air and an oxygen residue equal to one hundredth of the initial value.

Vacuum packaging effectiveness can be rendered useless by an inadequate choice of packaging material. Therefore, care needs to be taken when selecting a packaging material. In fact packaging materials are, in differing degrees, all permeable to gas and the vacuum of the package tends to accelerate the air return from the environment.

For long term preservation of the vacuum effects, “barrier” materials are needed to impede the entrance of oxygen for the time required. It is worth noting that the thicker the material, the slower (proportionally) the entrance of oxygen will be. Also, the less package surface area there is, there will be a proportionally slower permeation of gas. Finally, the entrance of the oxygen has different speeds as a function of the temperature and the lower it is, the slower the phenomenon. Vacuum forces a flexible packaging material to conform to the product shape. Once air has been replaced or eliminated from the package, there must be an adequate oxygen barrier and seal integrity to keep a low oxygen concentration inside the pack. Otherwise, the driving force created by the oxygen partial pressure differences (21% outside the bag and 0-2% inside the bag) will cause an ingress of oxygen and destroy the benefit of removing it in the first place. OTR values are used to compare the relative oxygen barrier capabilities of packaging films. An industry rule-of-thumb is that a material is considered a "high oxygen barrier" if its OTR is less than 1 cc/100 in^2/24 hr (15.5 cc/m^2/24 hr) (Hui, 2005).

Oxygen levels can be reduced by methods such as vacuum packaging, N_2 flushing or by the more recently developed approach of using O_2 absorbers or scavengers. Nitrogen flushing generally reduces the oxygen to 2 to 5% (Warmbier and Wolf, 1976),

which is not enough to prevent oxidation (Bishov et al., 1971, Kacyn et al., 1983, Labuza, 1971). Therefore the degree of the vacuum applied can have an important part in the effectiveness of vacuum packaging to prevent lipid oxidation.

Oxygen absorbers, a more recently developed approach to reduce oxygen levels, generally lower oxygen to less than 1%, and research has shown them to be effective in delaying oxidation in low-moisture foods (Berenzon and Saguy, 1998, Chan et al., 1993, Emenhiser et al., 1999, Ribeiro et al., 1993).

Light

The rate of lipid oxidation can be greatly influenced by light, which has created a serious problem for the dairy industry (e.g. fluid milk in untilted glass or plastic gallons), because of the development of off-flavors, a decrease in nutritional quality, and the severity and speed at which these phenomena develop (Bossett et al., 1994, Mestdagh et al., 2005)

Most ultraviolet (UV) light damage to lipids occurs at wavelengths less than 200 nm. Although UV light is thermodynamically capable of producing radicals directly in lipids, the process is not a competitive reaction. The principal light-absorbing groups of lipids are double bonds, peroxide O–O bonds, and carbonyls; the last two are most important (Schaich, 2005).

It is well known that exposure of foods and beverages to light may result in oxidation of lipids and other constituents, leading to the formation of off-favors, discoloration, and loss of vitamins, especially riboflavin and β-carotene. The effect of light on lipid oxidation and flavor stability of a particular food can be explained by both photolytic auto-oxidation and photosensitized oxidation (Bradley and Min, 1992).

Dairy products in particular are very sensitive to light oxidation because of the presence of riboflavin (vitamin B_2). This strong photosensitizer is able to absorb visible and UV light and transfer this energy into highly reactive forms of O_2 such as singlet O_2 (Min and Boff, 2002).

Important factors influencing the deteriorative effect of light are: the intensity and spectrum of the light source, the duration of light exposure, and the light transmittance of the packaging material (Bradley and Min, 1992).

Sattar et al. (1976) investigated the effect of light on the oxidation of milk fat and found that although there was an induction period for light-induced oxidation of milk fat, there was not for light-induced oxidation of vegetable oils. It was suggested that the induction period was due to the presence of α-carotene acting as a built-in light filter. Even though the presence of α-carotene in milk fat slowed down the rate of oxidation at the beginning of the trial, the light-exposed samples still showed a much higher oxidation rate than the samples kept in the dark. Packaging material plays an important role in protecting dairy powders against lipid oxidation induced by UV light. Therefore, the light transmittance of the packaging material has to be considered as an important factor, while suggesting a new packaging for dairy powders (Mestdagh et al., 2005).

2.11. Moisture Transfer and Shelf Life Stability of Dairy Powders

As explained in previous sections; powdery products become lumpy or cake, when they gain moisture. In addition, the moisture may lead to deleterious changes such as structural transformations, enzymic reactions, browning, and oxidation, depending on temperature and the availability of O_2 (Roos, 2001). Moisture or water vapor ingress in combination with light, O_2, and an elevated temperature can result in physical loss of

texture and caking due to lactose crystallization, microbial spoilage, nonenzymic reactions (such as Maillard browning), and fat oxidation (Uppu, 2002).

Although an $a_w < 0.6$ is considered sufficient to prevent the growth of microorganisms, chemical reactions and enzymic changes may occur at considerably lower levels (Roos, 2001). It is important for the determination of the maximum shelf life for milk powders (especially WMP) not to exceed a moisture content corresponding to an a_w at which the rate of lipid oxidation is at a minimum (Robertson, 2006b). Commonly the a_w of WMP varies from 0.25 (low) to 0.35 (high) (Baechler et al., 2005) and for SMP from 0.32 to 0.43 (Shrestha et al., 2008).

Moisture sorption isotherms (MSIs) for powders describe the equilibrium relationship between the moisture content of the powder and the relative humidity of the surrounding environment at a specific temperature. Such MSIs are major sources of information for optimizing concentration and dehydration processes, microbial growth conditions, and the physical and chemical stability of the product (Hardy et al., 2002) Knowing the MSIs of powdered milk products is essential to be able to predict their stability in association with packaging characteristics (Foster et al., 2005b). Figure 11 depicts a stability map for dairy powders containing amorphous lactose.

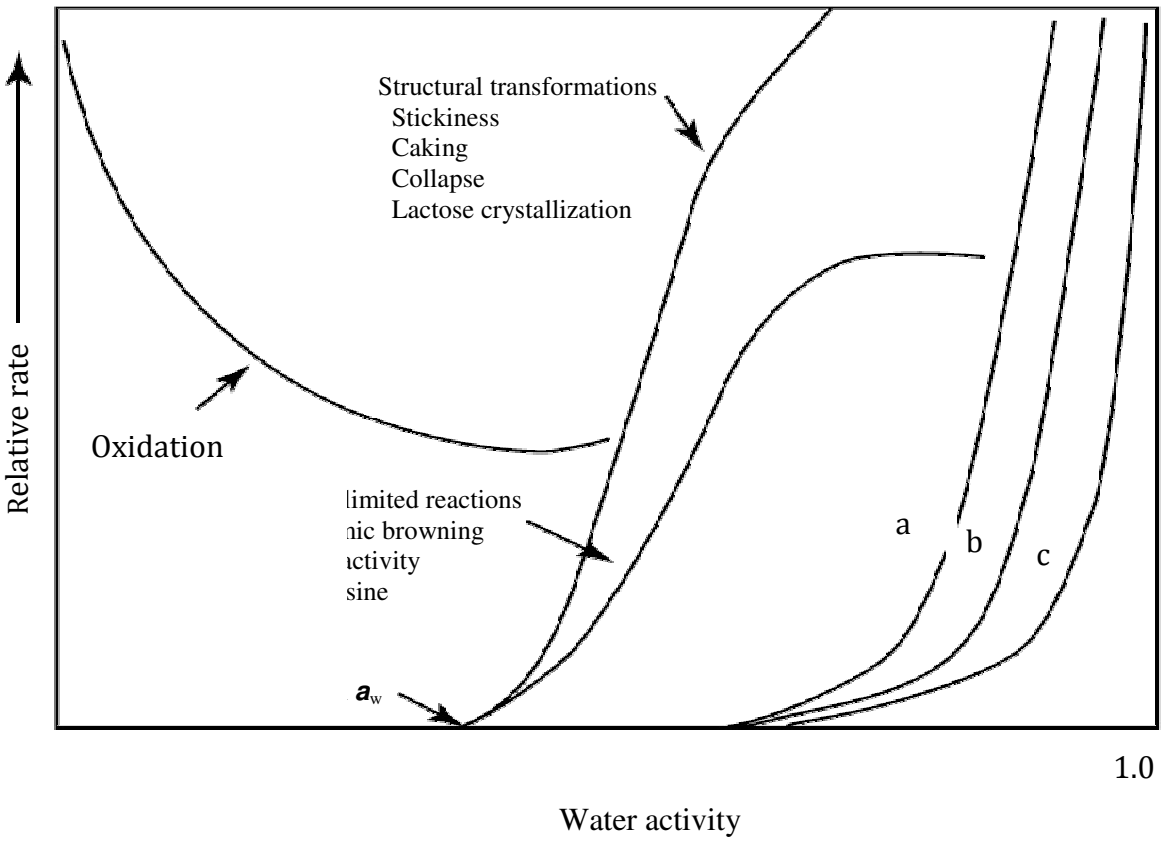

Figure 11: Stability map for dairy powders containing amorphous lactose. The critical water activity corresponds to the glass transition depression of amorphous lactose to 24ºC, which may enhance deteriorative changes and loss of quality (Roos, 2002).

a. Growth of molds, b. Growth of yeast, c. Growth of bacteria

Changes in the immediate environment (i.e., temperature, moisture, and gas composition) can cause different types of reactions that may be interrelated and sometimes act synergistically. Therefore, it is very difficult to control a particular reaction (Uppu, 2002).

Moisture content and a_w can often determine the rate of deteriorative reactions as well as microbial growth. As indicated earlier, prevention of microbial growth can be achieved provided $a_w < 0.6$ (Roos, 2001). However, increased moisture levels due to transmission or condensation of water vapor (due to temperature fluctuations) could

result in favorable conditions for microbial growth. Off-flavors, increased acidity, and visual and textural changes may be additional negative effects of microbial growth.

Protecting powder from moisture ingress, to preserve dairy powder characteristics, is one of the main purposes of packaging. The effectiveness of a package can be determined during shelf life testing or by combining information from break-point testing (holding at increasing humidities) and knowledge about the characteristics of the moisture permeability of the packaging material (Brown and Williams, 2003). Water vapor transmission rate (WVTR), is a measure of the passage of water vapor through a packaging material and it is defined as the steady state rate at which water vapor permeates through a film at specified conditions of temperature and relative humidity. Values are expressed in g/100 in^2/24 hr in US standard units and g/m^2/24 hr in metric (or SI) units. WVTR is the standard measurement by which films are compared for their ability to resist moisture transmission. Lower values indicate better moisture protection. Only values reported at the same temperature and humidity can be compared, because transmission rates are affected by both of these parameters (ASTM-E96).

In selecting a suitable packaging system for milk powders, three factors must be taken into account: (1) the initial moisture content of the powder, (2) the final acceptable moisture content of the powder, and (3) the required shelf life (Robertson, 2006b).

2.12. Significance of Packaging Material

In summary, the packaging of dairy powders needs to be considered in terms of its ability to block light, avoid transmission of water and water vapor, and prevent permeation of O_2. The fourth factor influencing the indices of failure of milk powder is the ambient temperature. Although temperature is a prime factor determining the shelf

life of milk powders, these products are not usually stored under controlled temperature. Therefore, storage of milk powders at high ambient temperature will accelerate deteriorative reactions, particularly if plastic barrier packaging materials are used, as the permeability of O_2 and water vapor increases at higher temperatures. In addition to fat oxidation, atmospheric O_2 and light are prime factors influencing the stability of vitamins A and D. These factors, in combination with environment factors such as temperature and moisture, influence the rate of reduction in the vitamin content (Ottaway, 1993).

A barrier can be defined in many ways depending on the desired level of protection from physical damage and chemical and biological changes that affect food quality and safety. A barrier is conceived to be for control of permeation of gases and vapor through the package. Barrier technology has been designed and developed for both flexible and rigid food containers. Different packaging materials have different barrier properties. A desired barrier level can be achieved by using one or more barrier materials for food packages, or by incorporating this barrier material using multi-layer structure, lamination, or coating techniques. Years of research and development have resulted in new barrier technologies for various foods and food products.

Table 2: Properties of selected food packaging materials (Fellows et al, 2002).

*= low **=medium ***=high
Thicker films of each type have better barrier properties than thinner films.
PVDC = polyvinylidene chloride

Film Type	Coating	Barrier to Moisture	Air/ Odors	Strength	Clarity	Normal Thickness Micrometers
Cellulose	-	*	***	*	***	21-40
Cellulose	PVDC	***	***	*	***	19-42
Cellulose	Aluminum	***	***	*	-	21-42
Cellulose	Nitro-cellulose	***	***	*	-	21-24
Polyethylene (low density)	-	**	*	**	*	25-200
Polyethylene (high density)	-	***	**	***	*	350-1000
Polypropylene	-	***	*	***	***	20-40
Polypropylene	PVDC	***	***	***	***	18-34
Polypropylene	Aluminum	***	***	***	-	20-30
Polyester		**	**	***	**	12-23
Polyester		***	***	***	**	-
Polyester		***	***	***	-	20-30

A study by Lim et al. (1994) on the effects of package type (Kraft paper/ nylon-polyethylene (PE), aluminum-PE (AL-PE) laminate) on the physicochemical changes during storage of whole milk powder (WMP) at 20 and 40°C showed that at 20°C, there was no significant change in the quality of powder stored in the different packages; powder packaged in Kraft paper/ nylon-PE under nitrogen or in AL-PE laminate packages under vacuum had lower peroxide and thiobarbituric values compared to the powder packaged in Kraft paper/ PE. Significant changes were noted in physic-chemical properties of WMP stored at 40°C. Comparing WMPs packaged in AL-PE laminate under nitrogen and vacuum, showed the least changes in physic-chemical properties. Kraft paper/ PE packaging was found to be inferior to all others.

The effect of plastic bags has been evaluated and compared to paper bags. Shakeel and Farkye, (2003) showed that plastic bags are similar to paper bags in maintaining milk powder quality during storage.

2.13. Packaging Related Problems Observed in Dairy Powders

Two packaging related problems are occasionally encountered in the handling and storage of 25 kg bags of dairy powders, one associated with production of low bulk density powder and the other associated with excessive air entrapment in the bag.

Bags containing low bulk density powder are bigger in volume and thus are more problematic to transport, and take up more storage space. Bags with an air entrapment problem tend to be unstable when stacked because of the ability of air to move around within the sack when subjected to an external force. This is sometimes referred to as the “drunken bag” problem. These problems do not occur frequently; nevertheless, when

they occur they cause difficulty in handling and storage of product (Fitzpatrick and O'callaghan, 1996).

Fitzpatrick and O'callaghan, (1996) showed that low particle density is mainly due to the formation of vacuoles in the particle, as a result of air being incorporated into the concentrate during atomization. The major factors that affect this are: composition, total solids content, state of denaturation of the concentrate and drier operating conditions. They concluded that air entrapment is caused by aeration and decompaction of powder by powder transport and entrapment of headspace air prior to heat sealing. It is recommended to store freshly dried powder for 1-2 days because fresh powder is troublesome to de-aerate. It is desirable to let the powder settle in the hopper in order to allow the powder to de-aerate and compact. Less aeration of powder occurs when the powder is augured into the bottom of the bag as opposed to dropping the powder into the bag.

Vacuum packaging seems to offer a solution for most of the problems mentioned above as it increases the bulk density of the powder in the bag by removing most of the air entrapped between the particles and pushes the packaging material tightly to the surface of the powder. However, Robertson, (2006) reported a major technical problem associated with vacuum packaging of milk powder; removing air from the package without removing powder fines, which could damage the vacuum pump and contaminate the sealing area of the laminate bag. The other technical issue in vacuum packaging large bags of dairy powders might be the required time to remove most of the normally trapped air among fine particles. A speedy packaging process is always necessary to reduce the production cost, hence the final price of the powder being manufactured. The technical

issues mentioned above are likely of the major reasons that vacuum packaging has not been commonly practiced in dairy powders industry; although the cost of replacing a current packaging system with new vacuum packaging machines is a separately important reason. Therefore, vacuum packaging machines designed for dairy powders have to be capable of: (1) not allowing the powder fines to escape from the opening of the bags under the high vacuum pressure applied (2) sucking up the air from the powder in a reasonably short time.

2.14. Properties of Dry Dairy Ingredients

There are several properties of dairy powders which are important both to the manufactures and the end-users. These include: functional properties such as: solubility, viscosity, gelatin, foaming and so on, as well as physical properties such as; particle size, particle density, bulk density, tapped density, flowability, compressibility, color and so forth. The next sections would define some of these quality attributes explicitly.

2.14.1. Definition of Solubility

The term solubility is also used to describe the dispersing characteristics of dairy powders when reconstituted with water. Solubility can be determined in various ways. In all tests, powder is dissolved under standardized conditions (duration and intensity of stirring, concentration, and temperature), then the fraction that has not been dissolved is determined (e.g., volumetrically after centrifugation or by determination of dry matter). Often one refers to this as a "insolubility index" (Walstra et al., 2006).

2.14.2. Definition of Particle Size

Particle size is an important physical property of milk powder and can relate to its appearance, reconstitution and flow characteristics. Particle size can be influenced by the milk characteristics, processing conditions and the type of equipment used in the drying process. For example, higher total solids in the condensed milk causes a larger particle size while a low concentrate viscosity reduces particle size. Atomization parameters influence product shape and size distribution of powder particles making it possible to control powder particle size. A spray-dried particle using centrifugal atomization will give a larger particle than the pressure nozzle atomization (Singh and Newstead, 1992).

Spray-dried powder particles usually are spherical with diameters in the range of 10-250 μm. Rapid dispersion requires a particle size of approximately 150-200 μm diameter (Caric, 1994).

Particle size, as an independent property is useless because there is no particulate material having a single particle size. Any powder would consist of a population of particles of the same chemical composition, but with a wide range of individual sizes Segregation will happen in a free flowing powder mixture because of the differences in particle sizes (Barbosa-Canovas et al., 1985).

In particle size measurement two important decisions have to be made before a technique is to be selected for the analysis; these are concerned with two variables measured, the size of the particle and occurrence of such size. The occurrence of amount of particle matter belonging to specified sizes may be classified or arranged by diverse criteria as to obtain tables or graphs (Ortega-Rivas, 2008).

2.14.2.1. Effect of Particle Size on Other Properties of Dairy Powders

The bulk density, compressibility and flowability of a food powder are highly dependent on particle size and its distribution (Peleg, 1977; Barbosa-Canovas et al., 1985).

Particle size in dairy powders can also affect powder reconstitution properties. Difference in particle size can lead to stratification of the powder with the higher solids concentrated at the top which will affect reconstitution of the dry product. Wettability and dispersibility of milk powder can also be influenced by particle size. Small particle size and symmetrical shape enhance close packing of particles and thus inhibit penetration of water. Larger particles more irregular in shape provide more space in the interstices for wetting. Generally large particles of dry milks exhibit good dispersibility. Dispersibility decreases as percentage of fine particles below 90 μm increases (Singh and Newstead, 1992).

Moreover, flowability also depends on particle size and shape. Large particles tend to flow more easily than smaller particles. Shape and size of the particles will affect the closeness of the particle pack, which in turn will affect the powder bulk density (Singh and Newstead, 1992).

2.14.2.2. Particle-size analysis

Coulter Laser light Scattering (LS) -230

In the past two decades, laser diffraction techniques have emerged as a replacement for traditional sieving and sedimentation methods for classifying sizes of particles in solution (Dufour et al., 1993, Kippax, 2005). The dairy industry has embraced

such tools, as the particle size of various components is known to influence the textural and sensory properties of dairy products (Instruments, 2005).

Modern laser diffraction instruments, such as the Coulter LS-230, are based on the Mie theory of light scattering, and contain laser instrumentation that is combined with non-laser light sources to obtain information about submicron-size particles (Kippax, 2010). During sample analysis, the light source generating a monochromatic beam is conditioned and focused to illuminate the particles traveling through the sample cell. Those particles scatter light and generate a unique scattering pattern depending upon the size distribution of particles present. The patterns are then Fourier transformed into an intensity pattern, which is measured using a photodetector array. This photocurrent is processed, digitized, and transferred to computer software, which applies the appropriate mathematical transformation based upon the light scattering theory selected. The resulting data is presented as a particle size distribution (Jillavenkatesa et al., 2001).

2.14.3. Definition of Density

The density of an object is defined as its total mass divided by its total volume. Determining the mass of an object is rather straightforward; it is the determination of volume that conceals the difficulty (Webb, 2001). The ‘volume’ of a solid object, whether a single piece or a mass of finely divided powder, is one of those concepts that can’t be bundled up into a single, neat definition. One must consult a particle technology’s lexicon to appreciate the various conditions under which volume is defined. Two sources for these definitions are the British Standards Institute (BSI) and the American Society for Testing and Materials (ASTM). Here one finds that the ‘volume’ of

a material is the summation of several rigorously defined elemental volumes (Webb, 2001).

A common masonry brick will serve as a good example of an object that contains all types of elemental volumes and differs in material volume according to the measurement technique, measurement method, and conditions under which the measurements are performed. A brick obviously is composed of solid material and it has a volume that can be calculated after measuring its length, width, and thickness. However, it also contains surface irregularities, small fractures, fissures, and pores that both communicate with the surface and that are isolated within the structure. Voids that connect to the surface are referred to as open pores; interior voids inaccessible from the surface are called closed or blind pores (Webb, 2001).

Surface irregularities compose another type of void volume. For example, assume the bulk volume of the brick is determined from linear measurements of its length, width, and thickness. It generally is understood that the value of volume determined in this way is limited in accuracy because the surfaces are not perfect. If a perfect plane were to be laid on one of the surfaces, there would be many voids sandwiched between the two surfaces. For lack of a standard definition, this will be referred to as 'external void volume' and will refer to the void volume between solid surface and that of a closely fitting envelope surrounding the object. It does not include pores that penetrate the interior of the particle. The meaning of the term is admittedly vague, but this volume can be determined or, at least, estimated under certain analytical conditions and can provide an indication of surface roughness (Webb, 2001).

When a solid material is in granular or powdered form (e.g. dairy powders), the bulk contains another type of void: interparticle space. The total volume of interparticle voids depends on the size and shape of the individual particles and how well the particles are packed.

Table 3: Definitions of various types of volumes. BSI = British Standards Institute, ASTM = American Society for Testing and Materials (Webb, 2001)

Volume Definitions	Volumes Included in Definition				
	Solid Material Volume	Open Pore Volume	Closed Pore Volume	Inter-particulate Void Vol.	External Void Volume
Absolute powder volume: (also called Absolute volume): The volume of the solid matter after exclusion of all the spaces (pores and voids) (BSI)	X				
Apparent particle volume: The total volume of the particle, excluding open pores, but including closed pores (BSI).	X		X		
Apparent powder volume: The total volume of solid matter, open pores and closed pores and interstices (BSI).	X	X	X	X	
Bulk volume: The volumes of the solids in each piece, the voids within the pieces, and the voids among the pieces of the particular collection (implied by ASTM D3766).	X	X	X	X	X
Envelope volume: The external volume of a particle, powder, or monolith such as would be obtained by tightly shrinking a film to contain it (BSI). The sum of the volumes of the solid in each piece and the voids within each piece, that is, within close-fitting imaginary envelopes completely surrounding each piece (Implied by ASTM D3766; see Table 2).	X X	X X	X X	X	X X
Geometric volume: The volumes of a material calculated from measurements of its physical dimensions	X	X	X	X	X
Skeletal volume: The sum of the volumes of the solid material and closed (or blind) pores within the pieces (Implied by ASTM D3766).	X		X		
True volume: The sum of the volumes of the solid material and closed (or blind) pores within the pieces (	X				
Void: Space between particles in a bed (BSI)				X	

Table 4: Definitions of various types of densities that follow from the volume definitions of Table 1. BSI = British Standards Institute, ASTM = American Society for Testing and Materials (Webb, 2001)

Density Definitions	Volumes Included in Definition				
	Solid Material Volume	Open Pore Volume	Closed Pore Volume	Inter-particulate Void Vol.	External Void Volume
Absolute powder density: The mass of powder per unit of absolute volume (BSI).	X				
Apparent particle density: The mass of a particle divided by its apparent (particle) volume (BSI).	X		X		
Apparent powder density: The mass of a powder divided by its apparent volume (BSI).	X	X	X	X	
Bulk density: (also called Bulk powder density): The apparent powder density under defined conditions (BSI).					
The mass of the particles divided by the volume they occupy that includes the space between the particles (ASTM D5004).	X	X	X	X	
The ratio of the mass of a collection of discrete pieces of solid material to the sum of the volumes of: the solids in each piece, the voids within the pieces, and the voids among the pieces of the particular collection (ASTM D3766).	X	X	X	X	
Effective particle density: The mass of a particle divided by its volume including open pores and closed pores (BSI).	X	X	X		
Envelope density: The ratio of the mass of a particle to the sum of the volumes of: the solid in each piece and the voids within each piece, that is, within close-fitting imaginary envelopes completely surrounding each piece (ASTM D3766).	X	X	X	X	X
The ratio of the mass of a particle to the envelope volume of the particle (implied by BSI).	X	X	X		X
Skeletal density: The ratio of the mass of discrete pieces of solid material to the sum of the volumes of: the solid material in the pieces and closed (or blind) pores within the pieces (ASTM D3766).	X		X		
Tap density (also called Tap powder density): The apparent powder density obtained under stated conditions of tapping (BSI).	X	X	X	X	
Theoretical density: The ratio of the mass of a collection of discrete pieces of solid material to the sum of the volumes of said pieces, the solid material having an ideal regular arrangement at the atomic level (ASTM).	X				
True density (also called True particle density); The mass of a particle divided by its volume, excluding open pores and closed pores (BSI).	X				

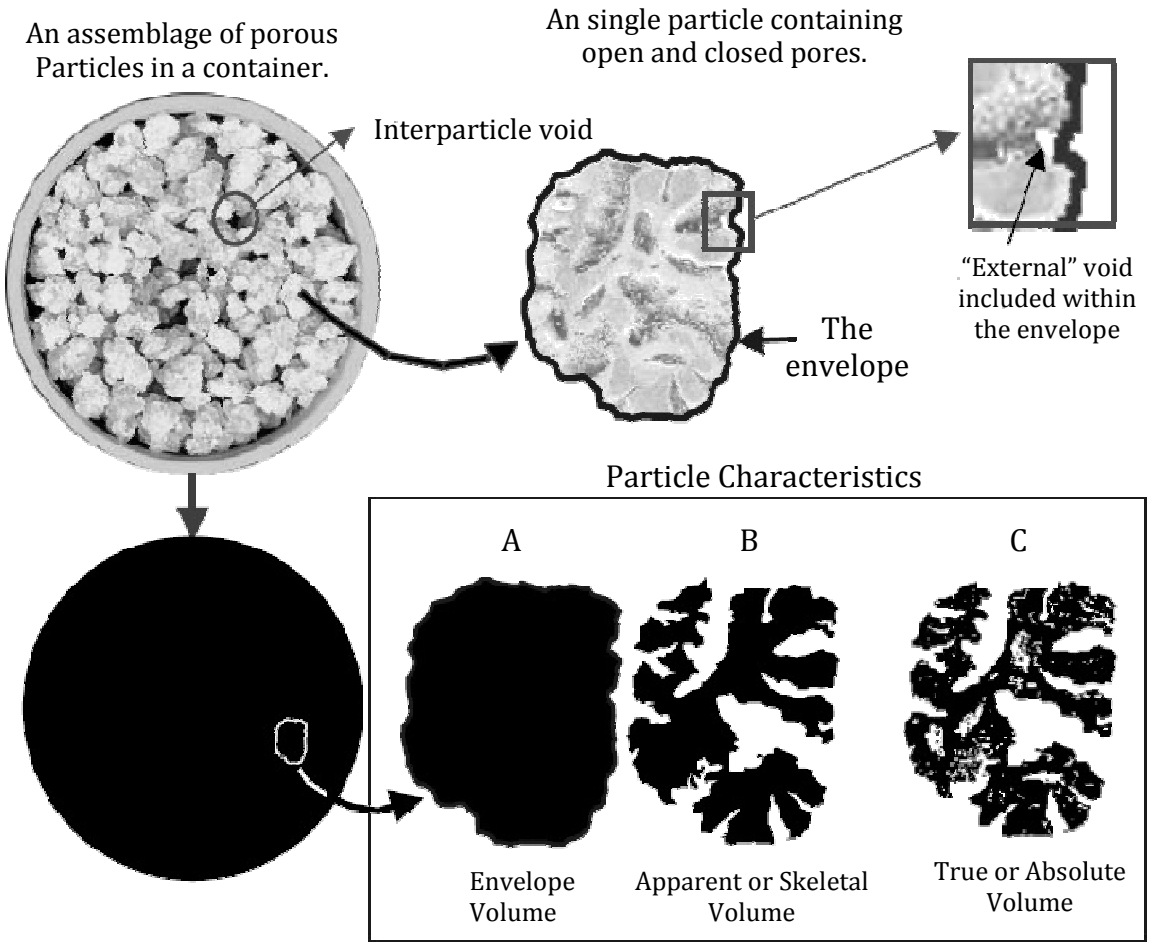

Figure 12: Illustration of various volume types. At the top left is a container of individual particles illustrating the characteristics of bulk volume in which antiparticle and "external" voids are included. At the top right is a single porous particle from the bulk. The particle cross-section is shown surrounded by an enveloping band. In the illustrations at the bottom, black areas shown are analogous to volume. The three illustrations at the right represent the particle. Illustration A is the volume within the envelope, B is the same volume minus the "external" volume and volume of open pores, and C is the volume within the envelope minus both open and closed pores (Webb, 2001).

Three volume definitions, those of apparent powder volume, bulk volume and envelope volume, have subtle differences. Apparent powder volume is most rigidly defined. It is the sum total of the four volumes indicated by the column headings in Table 3 (Webb, 2001). The difference between envelope and bulk volumes often is unclear. As can be seen in Table 3, ASTM's definition of envelope volume must be inferred from their definition of envelope density in table 4. It implies that the definition pertains only to a single particle, while BSI's definition encompasses a particle or monolith (singular implied), and a powder (by definition, a collection of fine particles) (Webb, 2001).

Density is considered quite relevant for determining other particle properties such as bulk powder structure and particle size; so it requires careful definition (Okuyama and Kousaka, 1991). Depending on how the total volume is measured, different definitions of particle density can be given: The true particle density, the apparent particle density, or the effective (or aerodynamic) particle density. Since particles usually contain cracks, flaws, hollows, and closed pores, it follows that all of these definitions may be different (Barbosa-Canovas et al., 2005b).

2.14.3.1. True Particle Density

True particle density represents the mass of the particle divided by its volume excluding open and closed pores, and is the density of the solid material of which the particle is made (Barbosa-Canovas et al., 2005b).

For pure chemical substances, organic or inorganic, this is the density quoted in reference books with physical/chemical data. Since most inorganic materials consist of rigid particles, while most organic substances are normally soft, porous particles, true

density of dairy powders is considerably lower than that of mineral and metallic powders. (Barbosa-Canovas et al., 2005b).

2.14.3.2. Apparent Particle Density

Apparent particle density is defined as the mass of a particle divided by its volume, excluding only the open pores, and is measured by gas or liquid displacement methods such as liquid or air pycnometry (Barbosa-Canovas et al., 2005b).

This density is important as a factor in predicting the solubility of a powder in water, how easily the powder particles sink and get dispersed in the water. The higher value of apparent density indicates the less number of closed pores in the particle, and this may influence the particle's resistance against water penetration.

2.14.3.3. Effective Particle Density

Effective particle density refers to the mass of a particle divided by its volume, including both open and closed pores. In this case, the volume is within an aerodynamic envelope as "seen" by a gas flowing past the particle. This density is of primary importance in applications involving bulk flow of air around particles like in fluidization, of liquid as sedimentation, or flow through packed bed (Barbosa-Canovas et al., 2005b).

The three particle densities defined above should not be confused with bulk density of materials, which includes the voids between the particles in the volume measured.

2.14.4. Definition of Bulk and Tapped Densities

An important quality attribute of milk powder is the bulk density. The bulk density is governed chiefly by the total solids of the feed to the atomizer, but also by the

temperature of the drying air. Bulk density is of considerable interest from an economic point of view because it influences the cost of storage, packaging and transport. (Robertson, 2006a). The higher the density of a packaged product, the lower volume it occupies. This would save space in both storage and transportation and less packaging material is needed for packaging.

Definitions and relationships between different types of densities are still confusing, and differences among measuring techniques can lead to considerable errors when determining them (Fasina, 2007). Over the years, in order of increasing values, three classes of bulk density have become conventional: poured, aerated and tapped (Barbosa-Canovas and Juliano, 2005). Each of these depends on the treatment to which the sample was subjected, and although there is a move towards standard procedures, these are far from universally adopted. There is still some confusion in the open literature as to how terms are interpreted. Some consider the poured bulk density as loose bulk density, while others refer to it as apparent density. Aerated density can also be considered to be a quite confusing term. Strictly speaking, aerated should mean that the particles are separated from each other by a film of air and not being in direct contact with each other. Some authors interpret the term as meaning the bulk density after the powder has been aerated. Tapped density, the bulk density after a volume of powder has been tapped or vibrated under specific conditions, can also be regarded as compact density (Ortega-Rivas, 2008).

Density of milk powder depends on the amount of air in vacuoles within individual powder particles (occluded air) and amount of air entrapped between neighboring powder particles (interstitial air) (Pisecky, 1997).

2.14.5. Definition of Occluded Air

Occluded air is referred to the air in vacuoles within individual powder particles and it is defined as the difference between the volume of a given mass of particles and the volume of the same mass of air-free solids, expressed in ml/100g. Occluded air is one of the most important factors for controlling bulk density. The amount of occluded air depends on the heat treatment applied to the feed, the method of atomization, and outlet air temperature (Pisecky, 1997).

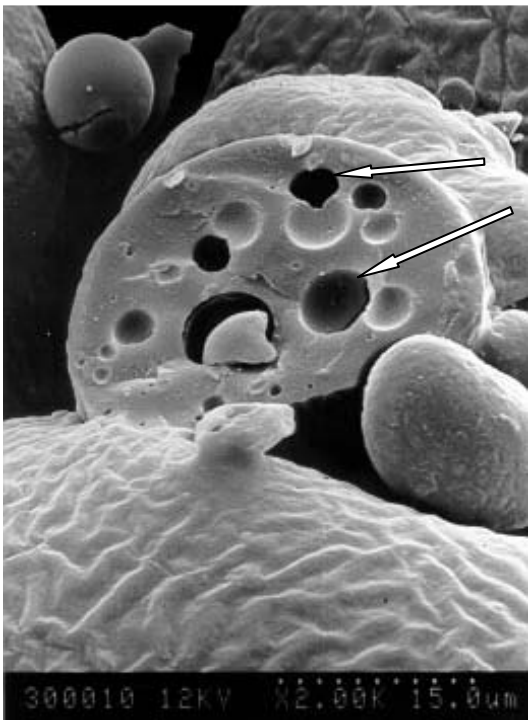

Figure 13: Electron microscopy photograph showing the internal porosity of milk powder particles (Hardy et al., 2002), Arrows point at vacuoles containg occluded air

Figure 13 shows the internal structure of a milk powder particle. The air entrapped in the vacuoles is called occluded air.

Occluded air content rises if air is incorporated into the feed prior to spray drying. Air is also drawn into the feed during atomization with rotating wheels. Air may be incorporated into the concentrate during transfer from the evaporator to the spray dryer or

during atomization. (Pisecky, 1997). The degree of denaturation of the whey proteins, concentration and temperature of the feed also has an effect on the amount of occluded air in the powder. Low heat products have a higher content of occluded air due to the higher content of nondenatured whey proteins which increases foaming properties. Therefore, the higher heat treatments have a higher bulk density due to a lower foaming ability and lower content of occluded air. During processing, the occluded air content can be minimized by heating the concentrate (50% total solids) up to 80°C (Pisecky, 1997).

Low bulk density can be achieved by increasing occluded air or by agglomeration. As occluded air content increases, particle volume increases, thus decreasing particle density and bulk density (Pisecky, 1997).

2.14.6. Definition of Interstitial Air

Interstitial air is referred to the air entrapped between neighboring powder particles and it is defined as the difference between the volume of a given mass of particles and the volume of the same mass of 100x tapped powder, expressed in ml/100g. This is a very complex property, too. The less interstitial air, the higher bulk density.

The amount of interstitial air is determined by the particle size distribution, surface geometry of particles, and the degree of agglomeration (Tamime, 2009). It may amount to 127 ml/ 100 g of dairy powder (alfa Laval/Tetra Pak, 1995). A powder with particles of the same diameter would be ideal from a drying point of view, but undesirable from a bulk density point of view, as the air space (the interstitial air) between the particles will be very large thus resulting in low bulk density (Tamime, 2009). The ideal is a wide particle size distribution with enough small particles to fill out the space between the medium and large particles thus resulting in a powder with high

bulk density. There is, however, a limit as to how small particles are wanted from a recovery point of view, plus the fact that a powder with many small particles will be dusty. Furthermore, they will affect the flowability negatively (Tamime, 2009).

A wider particle size distribution, but in the bigger particle size spectrum is therefore wanted. This can be obtained by using high solids content and/or viscosity, reducing the velocity of the wheel or pressure of the pressure nozzles, or using bigger nozzle size (Tamime, 2009). The result will however be very dubious in a single-stage dryer where the bigger particles call for higher outlet temperature thus increasing the occluded air content due to reasons already discussed (case hardening). Powders with extremely high bulk density can therefore only be achieved in two-stage dryers (Tamime, 2009).

It is the shape, as well as the size of the particle that will affect how close the particles are packed together, thus influencing bulk density. Spherical shaped particles make for a low content of interstitial air which consequentially results in higher bulk density. Irregularly shaped particles with attached smaller particles result in a lower bulk density.

The degree of agglomeration, as also mentioned earlier, is another factor governing the amount of interstitial air (Tamime, 2009). The powder leaving the chamber will be slightly agglomerated due to the primary agglomeration. In a one-stage dryer equipped with pneumatic conveying system, the problem does not occur due to the mechanical treatment it is exposed to. But in two-stage dryers the primary agglomeration is significant (Tamime, 2009). The agglomeration is developed due to the powder being more thermoplastic. As the mechanical treatment in the Vibro-Fluidizer is very gentle,

the agglomerates are not broken up. A pressure conveying system is therefore recommended, if a powder with very high density is wanted. It should however be pointed out that the primary agglomeration has a positive influence on the flowability of the powder (Tamime, 2009).

It has been observed that freshly made powder often exhibits low bulk density which increases several days after the production. This is caused by the electrostatic charge of the powder making the particles stick together, forming "agglomerates". As the time passes the powder will lose the charge and behave normally. An effective earth connection of all parts of the drying equipment can to some extent solve this problem (Tamime, 2009). The drying process can be manipulated (e.g., by using multiple stages or by returning fines to the atomization zone) to increase the levels of occluded and interstitial air and instantize the powder (Kelly et al. 2003).

2.14.7. Definition of Flowability

Many dairy powders are cohesive and many industrial silos storing them have mechanical and pneumatic discharge aids to help prevent arching and ratholing in an effort to maintain consistent reliable flow (Fitzpatrick et al., 2007).

Powder flow is defined as the relative movement of a bulk of particles among neighboring particles or along the container wall surface (Peleg, 1977). The practical objective of powder flowability investigations is to provide both qualitative and quantitative knowledge of powder behavior, which can be used in equipment design and in equipment performance prediction (Sutton, 1976). The flow characteristics of powders are of great importance in many problems encountered in bulk material handling

processes, because the ease of powder conveying, blending and packaging depends on flow characteristics (Chen, 1994).

Some of the flow problems caused by fine particles are: particle bridging in the hopper, particle adhesion to the wall and formation of a dead zone in the particle flow, flushing of powder through a feeder or hopper outlet, and blockage of hoppers and pipelines (Iinoya et al., 1988). In addition, since dairy powders are commonly used as ingredients to produce other food and beverage products, achieving a constant high quality in the end product requires a well-controlled flow rate of the dairy powders during the process.

In order to flow, powders must fail and their strength must be less than the load put on them. It is important to take into account the state of compaction of powders, as this strongly affects their flowability unless the powder is non-cohesive, like dry sand, and it gains no strength on compression. Flowability may also be strongly affected by humidity and, especially temperature. The time of consolidation can also have an effect on flowability of powders. Therefore flowability has to be tested under controlled conditions using sealed powder samples or air conditioned rooms or enclosures (Barbosa-Canovas et al., 2005a).

In industrial processes the terms cohesion and flowability are in common use, the former being, roughly, the resistance of the powder to flow and some researchers prefer to use the inverse of cohesion as a measure of a powder's flowability. Geldart et al., (2006) showed that measurement of poured angle of repose in an even simpler standardized tester gives values of flowability that agree well with those obtained from the inverse of cohesion deduced from well-known shear testers. They also concluded that while measurements of shear strength are essential for the proper design of storage vessels, for

the purpose of monitoring the flow behavior of bulk powders on a day-to-day basis, it is simpler and quicker to measure powder flowability more directly using angle of repose according to a standard technique.

Flowability is the ability of granular solids and powders to flow. Flow behavior is multidimensional in nature, and it depends on many physical characteristics. Flowability, in fact, is a consequence of the combination of the physical properties of material that influence flow, environmental conditions, and the equipment used for handling, storing, and processing these materials (Fitzpatrick et al., 2004). The capability of predicting powder flowability is helpful for preventing production stoppages in all bulk solid handling. However, no single test can fully quantify the flowability of a given powder.

Some of the factors that affect the flowability of bulk solids and powders include particle size, moisture content, humidity, flow agents, temperature, and pressure. Particle size and the particle-size distribution both play significant roles in flowability as do other properties, such as bulk density, angle of repose, and compressibility of bulk solids.

The angle of repose (AOR) has proved to be a useful property for characterizing the flow of powders; although, it can be used as an indirect indicator of flowability. Erica et al. (2009), in a study of static Angle Of Repose (AOR) observed a decrease in the value of the flowability rating with an increase in mean particle size, indicating an increase in flowability. Abdullah et al. (2010) suggested that an AOR below 30° indicates good flowability, 30-45° some cohesiveness, and 45-55° very cohesive (Carr, 1965), while Geldart *et al.* (2006) suggested the use of a 40° criterion in classifying free-flowing and cohesive powders.

2.14.8. Definition of Compressibility

Compression means a reduction in the bulk volume of the material as a result of displacement of the gaseous phase. Consolidation is an increase in the mechanical force of the material resulting from (interparticulate) particle-particle interactions (Lachman et al., 1986, Lannes and Medeiros, 2003). In fact, compressibility of a powder is a measure of its particle strength, which depends on its form and shape. Compressibility also determines the powder's flow properties. When a mass of particles is compressed, the voids between particles are reduced or eliminated and the powder tends to become a solid mass with fixed shape. Free-flowing powders are less compressible while nonfree-flowing powders are more (Onwulata, 2005).

2.14.8.1. Mechanisms of powder compaction

It has been proven that in fine powders the bulk compression takes place in two stages; the first stage of the compression process involves the movement of particles toward filling voids similar to or larger in size than the particles themselves. The packing characteristics of particles or a high interparticulate friction between them will prevent any further interparticulate movement. (Nystrom and Karehill, 1996). The second stage involves filling of smaller voids by particles that are deformed either elastically (reversible deformation) and/or plastically (irreversible deformation), and eventually broken down (Kurup et al., 1978; Carstensen et al.,1985; Duberg and Nystrom., 1986).

In fact, when a particulate solid is placed under pressure, a reduction in volume will occur due to the following mechanisms (Figure 14):

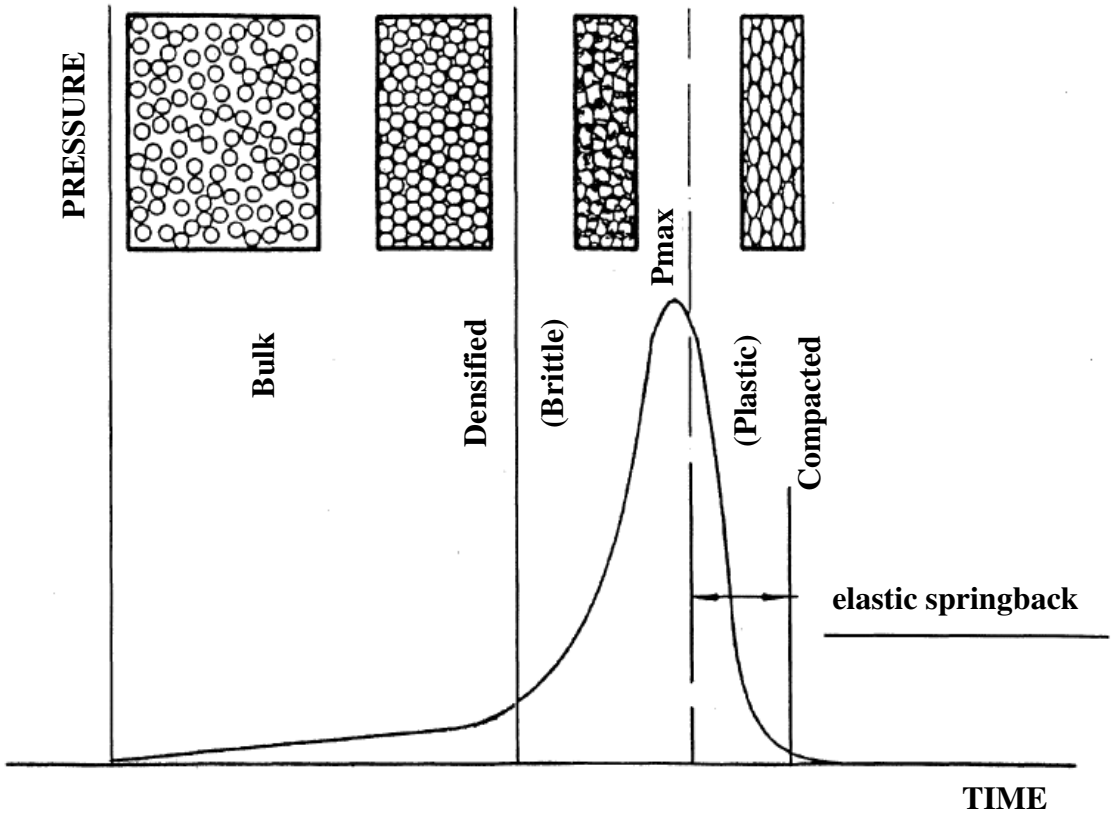

Figure 14: Mechanisms of powder compaction (Fayed and Otten, 1997)

1. At low pressure, rearrangement of the particles takes place, leading to a closer packing. At this stage, energy is dissipated mainly in overcoming particle friction, and the magnitude of the effect depends on the coefficient of interparticle friction (Fayed and Otten, 1997).

2. At higher pressures, elastic and plastic deformation of the particles may occur, causing particles to flow into void spaces and increasing the area of interparticle contact. Interlocking of particles may also occur. For materials of low thermal conductivity and low melting point, the heat generated at points of contact may be sufficient to raise the local temperatures to a point where increased plasticity and even melting facilitate particle deformation. With brittle materials, the stress applied at interparticle contacts

may cause particle fracture followed by rearrangement of the fragment to give a reduced volume (Fayed and Otten, 1997).

3. High pressure continues until the compact density approaches the true density of the material. Elastic compression of the particles and entrapped air will be present at all stages of the compaction process (Fayed and Otten, 1997).

The mechanisms discussed may occur simultaneously. The relative importance of the various mechanisms and the order in which they occur depend on the properties of the particles and on the speed of applying pressure (Fayed and Otten, 1997).

In vacuum packaging dairy powders, compaction occurs as the interstitial air is removed and small particles are brought into close contact. Ideally, the aim of vacuum packaging is to only densify the powder without any plastic (permanent) deformation and/ or fracture of particles, as plastic deformation or fracture of the particles might result in deterioration of powder quality (e.g. loss of solubility, flowability,…). Therefore it is worth detecting and quantifying any changes in quality attributes (densities, compressibility, flowability, particle size, solubility, etc.) of vacuum packaged dairy powders.

2.14.8.2. Compression Tests

Compression tests have been used widely in food powders, as a simple and convenient technique to measure physical properties such as compressibility and flowability. In order to get the pressure-density relationship for a given powder, a set of compression cells (usually a piston in a cylinder) is used. The tested powder is poured into the cylinder and compressed with a piston attached to the cross-head of a TA-XT2 Texture Analyzer or Instron Universal Testing Machine. Normally, a force-distance

relationship during a compression test will be recorded by the instrument. The details of the method would be explained in the next chapter. Generally, the higher the compressibility, the poorer the flowability (Schubert, 1987).

2.14.9. Definition of Surface Morphology

A milk powder particle generally consists of a continuous mass of amorphous lactose and other components in which fat globules, casein micelles, and serum proteins are embedded. The particles also contain vacuoles of occluded air where particle surfaces are not in contact. The surface of spray-dried particles is usually smooth but also may be wrinkled. Conditions such as higher inlet air temperature and larger temperature differences between the hot air and powder particles may contribute to wrinkles (Caric, 1994). The presence of particles of different morphology in the same sample can be attributed to the different drying conditions to which the individual particles were exposed.

2.14.9.1. Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) is recognized as unique tool in the visual examination of particles and their surfaces. The resolution is of the order of nanometers (magnifications in the range 20 - 100,000×). A fine beam of electrons of medium energy (5–50 keV) scans a gold-palladium coated sample producing secondary electrons, backscattered electrons, light or cathodoluminescence and X rays. SEM is routinely used for imaging particles in the micron and smaller size range and for examining the surfaces of larger particles. The resolution allows identification of specific surface geometric features that are indicative of structural phenomena (Hickey et al., 2007).

The standard scanning electron microscope (SEM) operates with secondary electrons. This approach provides a topographical view of the specimen. At higher accelerating voltages in the SEM, the signal is a mixture of surface properties and, from a small volume under the surface as the accelerating voltage is decreased; the signal becomes more related to surface properties. While the sample generally needs to be coated with a conducting layer to prevent charging, it is possible to examine uncoated samples at an appropriately low accelerating voltage. However, at lower accelerating voltages fewer secondary electrons are generated, and this can lead to difficulties in imaging. Typically, this form of SEM is most useful for examining powder particles. Backscattered electrons provide information about the atomic number distribution in the specimen with areas of higher atomic number appearing brighter than those with lower atomic number (Tamime, 2007).

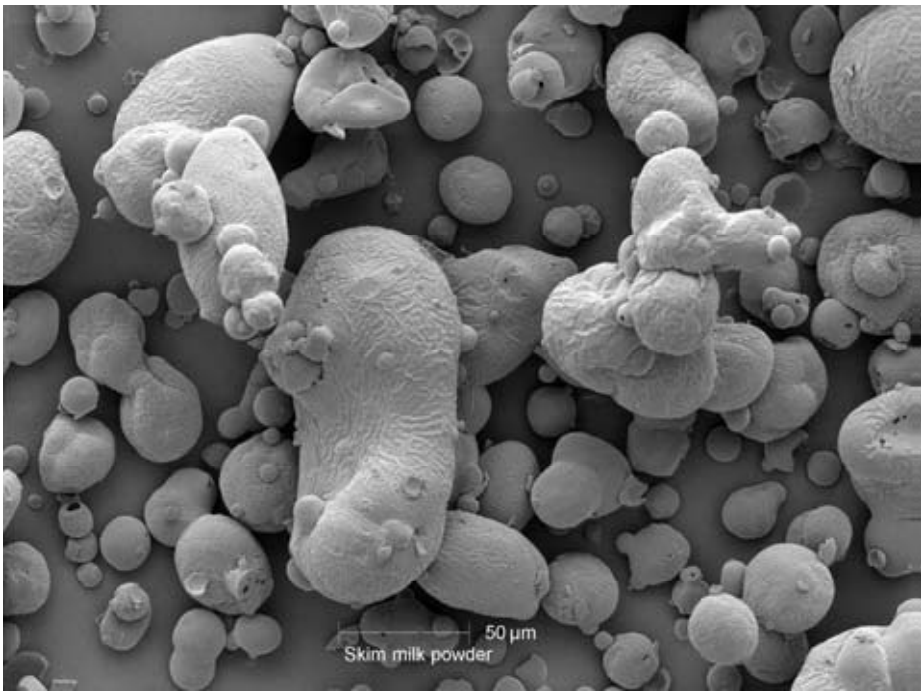

Figure 15: Scanning electron microscopy (SEM) of spray-dried skimmed milk powder (M. Kalab)

Scanning electron microscopy has been instrumental in elucidating the crystallization of lactose and its relationship to quality losses during the storage of milk powders. When milk is dried, amorphous lactose forms which is highly hygroscopic. Moisture uptake during storage induces the crystallization and cementing of powder particles into large lumps, a phenomenon known as caking.

Using scanning electron microscopy; Roetman, (1979) showed that lactose crystals present at the beginning of storage have the typical tomahawk shape and good keeping properties, while those formed during storage are needlelike and possess poor attributes. Saltmarch & Labuza, (1980) reported that shifting from amorphous to crystalline lactose releases water and causes microstructural collapse, favoring nonenzymatic browning of whey powders. El-Sayed et al., (1990); King, (1990); Hassan and Mumford, (1993) studied the effect of drying temperature on microstructure of milk powder and reported that increasing the drying temperature accelerates the drying rate of droplets, promoting the fast formation of the crust or skin. Piseckey (1978) and Charm (1981) have also demonstrated that, when the drying temperature is sufficiently high, moisture is evaporated very quickly and the skin becomes dry and hard, so that the hallow particle cannot deflate when vapor condenses within the vacuole as the particle moves into cooler regions of the drier. Birchal et al., (2005) reported that fast formation and expansion of the crust or skin can also damage the particle surface, creating fissures or breakages. However, when the drying temperature is lower, the skin remains moist and supple for longer so that the hollow particles can deflate and shivel as they cool. Rosenberg et al. (1988), Rosenberg and Young (1993) and Walton and Mumford (1999) have also observed similar phenomena.

The studies mentioned above and many more have used scanning electron microscopy as a tool mainly to link processing variables with the surface characteristics and morphology of milk powder particles. As vacuum packaged powder particles are kept under vacuum pressure over the storage time, they might go through elastic and/ or plastic deformation and/ or fracture, which might finally alter powder's quality attributes (e.g. particle size, solubility, flowability,…). Therefore, it is worth using scanning electron microscopy to detect any changes in surface morphology of vacuum packaged dairy powders.

2.14.10. Definition of Color

Color, an aspect of the appearance of food, is one of the attributes that affect the consumer perception of quality. The color changes in dairy powders during storage are reportedly caused due to a set of chemical reactions called Maillard reaction also referred to as non-enzymatic browning (Thomas et al., 2004b). Although color is usually indicative of degradation by Maillard reaction and nonenzymatic browning, it can also be useful in providing information about the degree of lipid oxidation (Laroche et al., 2005).

2.14.10.1. Color measurement

Methods which have been employed to determine browning reactions include chemical analyses and optical measurements, as well as visual examination (Morales and van Boekel, 1998). Colorimetry has also been employed by Morales and van Boekel (1998), Nielsen et al. (1997), Owens et al. (2001), Pagliarini et al. (1990), Rankin and Brewer (1998), Rhim et al. (1988), to measure color changes in dairy products.

One of the most common ways of evaluating color changes involves Hunter's values (L- a- and b-) (Phillips et al., 1995; Ameny and Wilson, 1997; Nielsen et al., 1997b). Hunter's L-value describes the lightness of a sample, from black (0) to white (100). A positive Hunter's a- value is indicative of a red tint, while a negative value implies green. In contrast, Hunter's b-value denotes a yellow hue when positive and a blue color when negative.

Triestimulus color measurement has been employed to measure color changes in dairy products by many researchers. Grigioni et al., (2007) studied the color changes of milk powder due to the thermal treatment applied to the milk (before drying step) and the season of manufacture. Evaluation of the color of WMP was carried out using a reflectance spectrophotometer (BYK Gadner Color View model 9000) according to CIE Lab scale. The whole milk powder obtained under indirect heat treatment conditions (IHT: 90-93 °C; 180 s) showed significant lower values of "L" than WMP elaborated with direct heat treatment (DHT: 105 °C; 30 s). WMP produced in summer revealed the lowest levels of "L" in contrast with WMP from autumn and winter. In general, The WMP elaborated in spring and summer showed higher levels of "b" for both thermal treatments. Rhim et al., (1988) evaluated tristimulus Hunter "L", "a" and "b" values of milk subjected to heat treatments over a wide range of temperatures (l00 -150°C) and times (0.5-180 min). According to their results, Hunter "L"values decreased following first-order kinetics (E_a = 114.8 KJ/mol) and Hunter "a" and "b"values increased following zero-order kinetics (E_a = 104.9 and 110.3 KJ/mol, respectively). The Browning Index, also (reflectance ratio of 520/430), correlated with milk browning effect. Its value increased following zero-order kinetics (E_a = 123.5 KI/mol).

Rankin and Brewer, (1998) compared nonfat milks inoculated and fermented with exopolysaccharide and non-exopolysaccharide producing cultures to nonfat, 2% milkfat and whole milk with instrumental color measurements. According to their results, L- and a- values for fermented nonfat milks were higher than nonfat milk. No differences in L-, a-, or b- values were found between milks fermented with exopolysaccharide as compared to non-exopolysaccharide producing strains.

Hunterlab Ultra Scan XE Spectrophotometer

The HunterLab Ultra-scan XE spectrophotometer is an instrument for physical analysis which provides wavelength by wavelength spectral analysis of the reflecting and/ or transmitting properties of objects without interpretation by a human. It can indirectly calculate psychophysical (colorimetric) information as tristimulus values; L, a, b, XYZ, etc. (Hunter and Harold, 1987). The L axis runs from top to bottom. The maximum for L is 100, which would be a perfect reflecting diffuser. The minimum for L would be zero, which would be black. The "a" and "b" axes have no specific numerical limits. Positive "a" is red. Negative "a" is green. Positive "b" is yellow. Negative "b" is blue. Below is a diagram of the Hunter Lab color space. There are delta values (ΔL, Δa, and Δb) associated with this color scale. These values indicate how much a standard and sample differ from one another in L, a, and b. The ΔL, Δa, and Δb values are often used for quality control or formula adjustment. Whether the sample is redder or greener than the standard, is indicated by the sign of the delta value. For example, if Δa is positive, the sample is redder than the standard (Billmeyer and Saltzman, 1981).

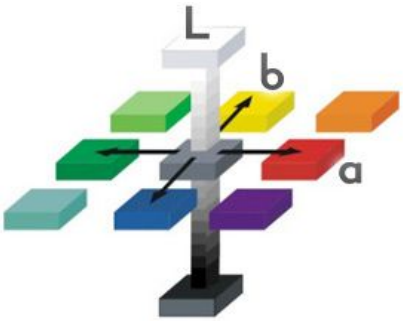

Figure 16: The Hunter Lab color scale

$\Delta L = L_{sample} - L_{standard}$

+ ΔL means sample is lighter than standard

- ΔL means sample is darker than standard

$\Delta a = a_{sample} - a_{standard}$

+ Δa means sample is redder than standard

- Δa means sample is greener than standard

$\Delta b = b_{sample} - b_{standard}$

+ Δb means sample is yellower than standard

- Δb means sample is bluer than standard.

2.15. Significance of packaging in relation to storage and production cost

The costs of warehousing, transportation and packaging material are of the important elements contributing to the final cost of all packaged food products. Selecting a packaging method that reduces these costs would reduce the final price of the food product being packaged (Saghir, 2004).

2.15.1. Storage considered as production cost

More compact packaging will result in a reduced material cost. The amount of warehouse space required is also reduced, further impacting logistics costs. By packaging with increased ease of handling, the savings can be extended, as less labor would be required. The domino effect continues through redesigning pallet configuration and shipping more products per pallet.

2.15.2. Packaging, the basis of the storage system

The dimensions of a package will affect its efficiency and ultimately the total cost of the product all throughout distribution. The cubic volume and dimensional layout of dairy powder packages need to be optimized for international and U.S distribution. Bick shaped vacuum packaged dairy powders are expected to allow accommodating more products per pallet, comparing to the pillow shaped atmospheric packaging currently used by dairy powder manufacturers. However, the efficiency of the two packaging methods is yet to be thoroughly investigated and compared.

2.15.3. Transport, an integral part of the storage and distribution

A thorough understanding of the distribution system is fundamental for designing cost-effective packaging that provides the appropriate degree of protection to the product and is acceptable to the user(s). Distribution may be defined as the journey of the pack from the point of filling to the point of end use (Robertson, 1990). The efficiency throughout distribution can come down to very simple geometry. The way products can be grouped, whether on a pallet, in a sea container or in the master pack, may ultimately determine their cost effectiveness. Cubic compact packages are likely to be more

efficiently grouped and stacked on pallets or in warehouse racks, comparing to packages with a large headspace and curved edges.

2.16. Packaging, Unitiziation, and Handling

2.16.1. Definition of Unit load

A number of items, or bulk material, so arranged or restrained that the mass can be picked up and moved as a single object, which is too large for manual handling, and will retain its initial arrangement for subsequent movement, upon being released (Tompkins et al., 1996).

The size and type of package determine the character of a unit load. The form of the packaging and unit load contribute to choice of storage function and its continual operating characteristics and potential efficiency. One example of this is polythene film, used to "shrink-wrap" pallet loads of dairy powders; which allows better use of space provided, more stable stacking and easier checking (Tompkins et al., 1996).

As unitized loads of dairy powder bags are generally of fairly large weight and volume, the method of handling them, i.e. how to hold, lift and carry them, can be an important issue. The common method of handling a unit load of dairy powder bags is putting a lifting device under the load, and then handling this device along with the load. The device is called pallet.

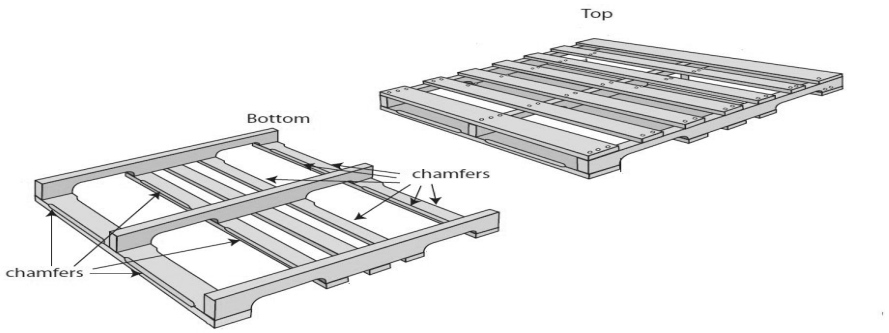

Figure 17: Top view (on the right) and the bottom view (on the left) of a wooden pallet

2.16.2. Definition of Pallet Pattern

Generally, same or similar items of fairly regular shape and size are put on a pallet for unitization. Items are generally kept in layers in definite arrangements. Placement of goods in such arrangements is called pallet pattern.

Figure 18 shows a few of these pallet patterns referred to as block, brick, row and pin-wheel. The (2 × 3) pattern is the common pallet pattern for dairy powders (Bush D., personal communication, Jan.12 2010). The choice of a particular pattern depends on a number of factors like pallet type and size; dimensions, shape, fragility, weight, container used etc. of the item; container dimension, shape, strength, amount of interlock etc.; handling equipment and attachments used; warehouse layout and type of movement.

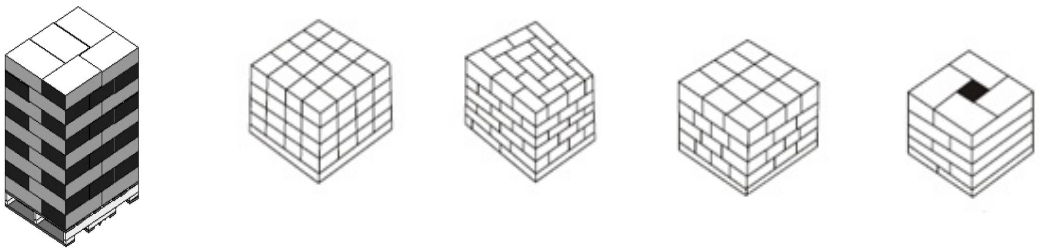

Figure 18: Different pallet patterns

2.17. Storage and Handling Advantages of Vacuum Packaged Dairy Powders

Product volume and density are of major factors affecting transportation costs of products. Transport cost per unit of weight decreases as load volume increases (as illustrated in Figure 19) (Bowersox et al., 2009). This occurs because the fixed costs of pickup, delivery, and administration can be spread over incremental volume. This relationship is limited by the size of the transportation vehicle. Once the vehicle is full, the relationship begins again for each additional vehicle (Bowersox et al., 2009). The management implication is that small loads should be consolidated into larger loads to maximize scale economies. Density is a combination of weight and volume. Weight and volume are important since transportation cost for any movement is usually quoted in dollars per unit of weight. Transport charges are commonly quoted as amount per hundred weight or centum weight (CWT) (Bowersox et al., 2009). In terms of weight and volume, vehicles are constrained more by cubic capacity than by weight. Since actual vehicle, labor, and fuel expenses are not dramatically influenced by weight, higher-density products allow relatively fixed transport costs to be spread across more weight. As a result, higher density products are typically assessed lower transport costs per unit

of weight. Transportation cost per unit of weight declines as product density increases (as illustrated in Figure 20). In general, it is ideal to improve product density so that trailer cubic capacity can be fully utilized (Bowersox et al., 2009). Vacuum packaging has a great potential in increasing density of products being packaged by removing air, and this effect can be more distinctive for products such as dairy powders because of the interstitial air entrapped in their particulate structure.

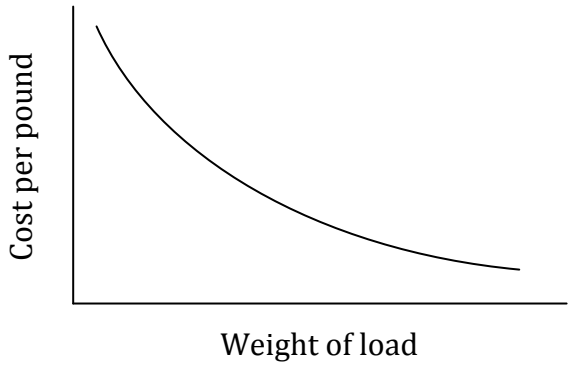

Figure 19: Generalized relationship between weight and transportation cost/ pound (Bowersox et al., 2009).

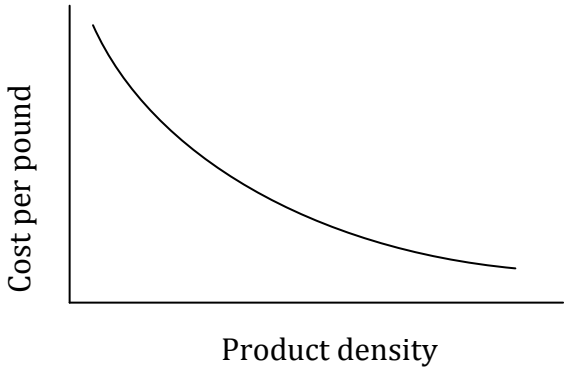

Figure 20: Generalized relationship between density and transportation cost/ pound (Bowersox et al., 2009).

Vacuum packaging dairy powders may result in a uniform shape of package. The effect of vacuum packaging on the shape of bags would be more profound if side gusseted bags are used. The more uniform shaped bags would be much easier to handle in bulk, better suits a mechanized storage system, and improve the efficiency of using the space on a pallet, in transportation and storage. Vacuum packaging saves space in a bag by removing the air from the headspace and the interparticulate space.

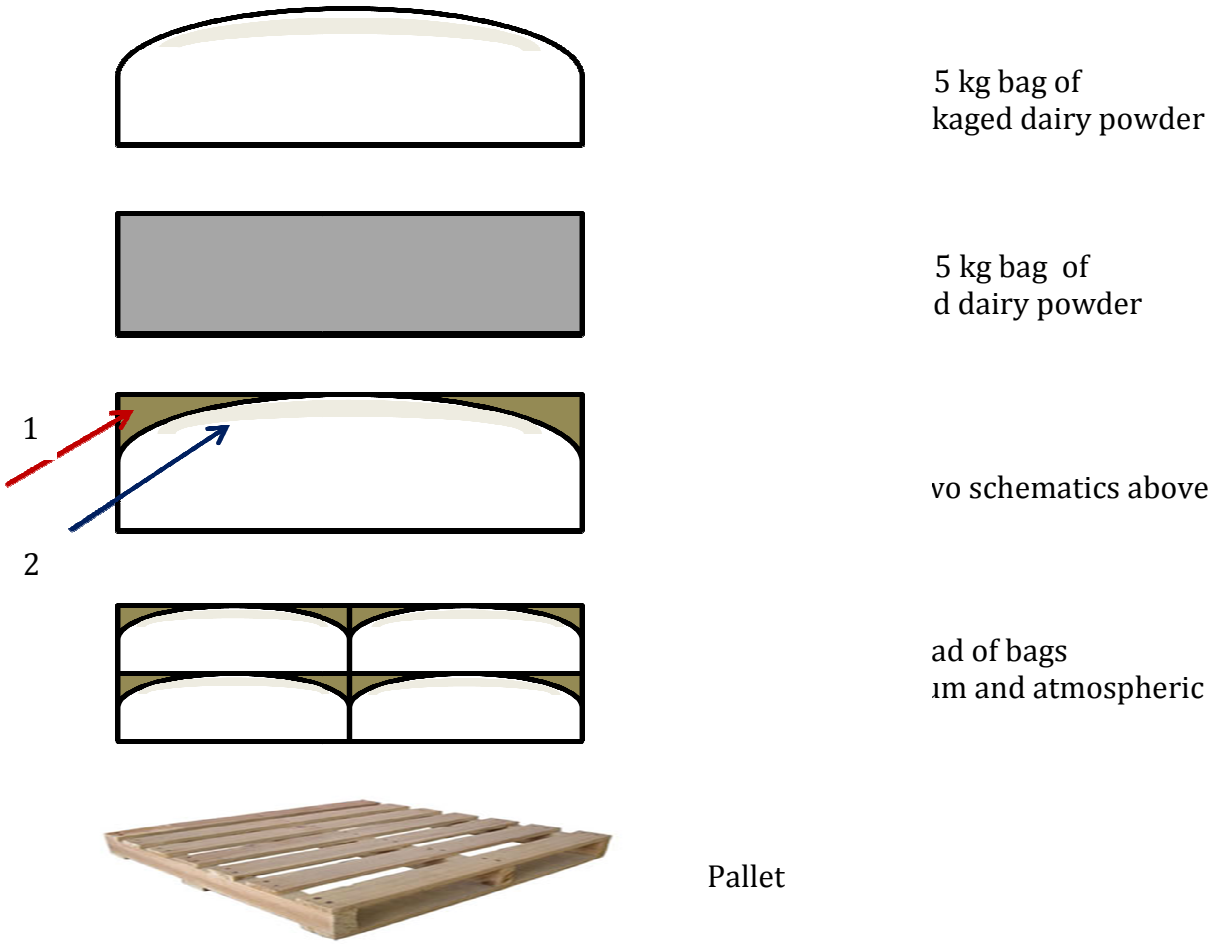

Figure 21: Schematic comparison of vacuum and atmospheric packaged bags of dairy powders. (1) The colored area in the overlap pictures shows the space that is wasted in atmospheric packaging and saved by vacuum packaging. (2) The headspace of the bag

2.18. Evaluation of storage and stacking properties

One key to evaluating packaging, as it affects distribution costs, is not overlooking dimensional alterations and options. An inch off of one dimension may allow the fill pattern to change and another entire row or another layer to be added. Determining the best pallet arrangement or pallet pattern and the best vehicle loading arrangement, and calculating the consequences of possible dimensional changes, is very difficult, if done by hand. Pallet patterns may be simple or very complex. To investigate and seek the optimal pattern, computer software, CAPE®, is available to take over the hard work (Goodwin and Young, 2010). In addition to just pallet patterns and vehicle loading, this software allows manipulation of package size or volume and estimation of stacking strength required, among other functions. Moreover, programs like CAPE® can be invaluable in evaluating the effect of dimensional options on container quantities quickly. When embarking on a redesign, manufacturers should first assess the efficiency of the current packaging to use as a baseline (Goodwin and Young, 2010). Packaging-specific software programs such as CAPE, TOPS, ArtiosCAD, and Solidworks can be used both to assess the efficiency of the current packaging and to design the most efficient new package. In this study, CAPE PACK v2.09 software was used to measure the efficiency of using the storage space.

2.19. Summary of Literature

Dry dairy ingredients can be defined as a range of powder products with varying bovine milk protein, fat, lactose and minerals concentrations, produced through different processes such as fat separation, acid/ enzyme coagulation, ultrafiltration of milk and subsequent water evaporation. The manipulation of processing parameters has allowed

manufacturers to create a wide variety of dry dairy ingredients with varying protein, lactose, fat, and mineral concentrations resulting in different functional and nutritional properties. There is a demand for more efficient packaging methods that not only keep the quality of dry dairy ingredients during the storage but also add to the efficiency of using the storage space and ease of handling. In-package oxygen level is shown to play a crucial role in the degradation of functional and sensory properties of dry dairy ingredients. Minimizing or removing oxygen from the package is shown to inhibit the chemical deterioration, specifically fat oxidation, of dairy powders in the package. Vacuum packaging can be an attractive method for keeping the quality of dry dairy ingredients and provides added value; because of the inherent compactness of these products. Vacuum packaged dry dairy ingredients may also have added ease of handling for end users. However little is known about the impact of vacuum packaging on the key properties of dry dairy ingredients such as density, flowability, compressibility and solubility. These properties might be correlated with vacuum pressure at different levels and/or different times during storage and these relationships would be well worth further investigation.

3. JUSTIFICATION, HYPOTHESIS, AND OBJECTIVES

Dry dairy ingredients can be defined as powder products with varying bovine milk protein, fat, lactose and mineral concentrations produced through different processes such as fat separation, acid/ enzyme coagulation, ultrafiltration of milk and subsequent water removal. Many dairy powders are often stored for a long period of time, up to 1 - 2 years. Unfortunately over the storage time many of these powders exhibit poor functional and sensory properties resulting in lower acceptance of these products in the market and financial losses for both the manufacturers and the consumers, consequently. An extended review of the scientific literature hints at several critical factors, including storage duration, temperature, the compositional properties of dairy powders, especially the fat content and in-package oxygen levels, that likely influence the functional and sensory properties of dairy powders. The in-package oxygen level is implicated as a critical factor in several studies (Andersson and Lingnert, 1998, Chan et al., 1993, Coulter, 1947, Min and Lindamood, 1989, Tuohy, 1984, Warmbier and Wolf, 1976), who collectively describe significant improvements in the sensory quality and shelf life of milk powders stored in the absence of oxygen. Although there has been a general consensus of opinion among investigators that reduced levels of oxygen in the package retard the development of oxidized flavor, especially in whole milk powders, no agreement exists as to the minimum levels needed during storage. For example, Lea et al., (1960) concluded that the development of oxidized flavors in spray-dried whole milk could be controlled by reducing the oxygen content in the free space of the container to 1 to 3%, while Coulter (1947) and Coulter et al. (1948) concluded that less than 1% oxygen in the packing gas was necessary to prevent oxidation of the whole milk powder, and

Shaffer, (1945) reported 3% as the upper limit of in package oxygen content for extended storage life at room temperature.

Despite the evidence of the effect of reduced in package oxygen level on the flavor of whole milk powder, there exists no study in the scientific literature examining the relationship between vacuum packaging and physical properties and solubility of dry dairy ingredients with different compositional and bulk properties. Additionally, there are no studies which link the level of vacuum and the powder type with changes in powder properties (bulk density, tapped density, particle density, particle size, surface morphology, flowability, compressibility and solubility) over the storage time. There is also no mention of a vacuum packaging method that is designed to improve efficiency of using the storage space in dry dairy ingredients, which may be a superior choice of packaging for the dairy powder manufacturer.

This thesis project examines the effect of vacuum packaging at different levels of vacuum on the bulk density, tapped density, particle density, particle size, surface morphology, flowability, compressibility, and solubility of 6 different types of dairy powders including; WMP, NFDM, BMP, SW, WPC80 and MPI over a 12 month storage period. The relationships between powder type, vacuum level and storage time on all the properties above will be examined. Once these relationships have been elucidated, steps will be taken to quantify the efficiency of the proposed vacuum packaging method in the use of storage space in comparison with the currently practiced atmospheric packaging method.

The hypotheses of this study were:

1. Vacuum packaging does not have any detrimental effect on the physical properties and solubility of dairy powders.
2. Vacuum packaging does save space in storage by increasing the bulk density of dairy powders.

The objectives of this study were:

1. To conduct initial feasibility tests of vacuum packaging dry dairy powders.
2. To characterize the effects of full or partial vacuum pressure (at three levels) on physical properties and solubility of six types of dairy powders: nonfat dry milk, whole milk, sweet whey, buttermilk, whey protein concentrate and milk protein isolate during a twelve month storage period.
3. To quantitatively compare the space saving effect of the proposed vacuum packaging method to the conventional atmospheric packaging both in a bag and on a pallet.
4. Prepare prototype samples of vacuum packaged powder for demonstration purposes.

4. MATERIALS AND METHODS

4.1. Materials

Six types of industrial spray-dried dairy powders (whole milk powder (WMP), buttermilk powder (BMP), nonfat dry milk (NFDM), sweet whey powder (SWP), whey protein concentrate-80 (WPC80) and milk protein isolate (MPI)) were selected based on certain criteria which mainly include specific compositional properties and their price. For example, high fat content (whole milk powder), low fat content (non fat dry milk), high protein content and price (whey protein concentrate 80 and milk protein isolate), high lactose content (sweet whey powder, non fat dry milk, buttermilk powder, whole milk powder). A 25 kg bag of each powder was obtained from different dairy companies in the United States. The powders were commercial products that had been freshly manufactured and packaged for consumer use.

Figure 22: Appearance of six different types of dairy powders used: 1. whole milk powder (WMP); 2. buttermilk powder (BMP); 3. nonfat dry milk (NFDM); 4. sweet whey powder (SWP); 5. whey protein concentrate80 (WPC80); 6. milk protein isolate (MPI)

The composition of the powders used is shown in Table 16. The protein, fat, moisture, and mineral contents were determined by Kjeldahl method, Mojonnier ether extraction method, and gravimetric methods, respectively. The lactose content was determined by difference. The bulk density, tapped density, particle density, particle size, compressibility, flowability, surface morphology and solubility were determined according to the methods explicitly explained in this chapter.

4.2. Vacuum Packaging

Each of the six commercial powders was repackaged under varying degrees of vacuum (1, 0.7, 0.4 bar) and a control with no vacuum (duplicate samples at each vacuum level). Then the vacuum packaged powders were stored (without stacking only in one

layer) at 25°C and 60% relative humidity, for 3, 6 and 12 months (separate samples for each of these three sampling times, according the experimental design shown in (Figure 25). The packaging material used was multiwall foil gusset bag (Stock Bag Depot, CA) with a three layer construction consisting of polyethylene terephthalate, aluminum and linear low density polyethylene (PET/AL/LLDPE) totaling approximately 135µ. The packaging device used was a modified MVS 38 vacuum sealer (Minipack America, Inc., Orange, CA). The bags were filled with powders using a metal spoon. Only 4/5 of the volume of each bag was filled with powder and then each bag was tapped manually 5 times on a benchtop. To have a stronger seal, after vacuum packaging all bags were resealed using an 8 inch. foot operated heat sealer (Stock Bag Depot, CA).

To keep into account the effect of weight, it was kept constant within each powder type.

Figure 23: MVS 38 vacuum sealer (left), multiwall foil gusset bag (right)

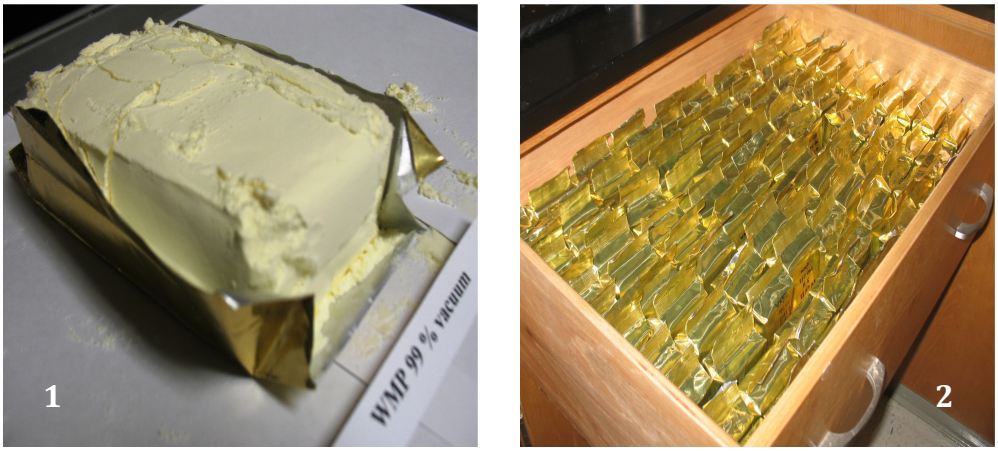

Figure 24: 1. Storage of vacuum packaged bags without any stacking; 2. Appearance of whole milk powder after 3 months storage under 1 bar vacuum pressure

4.2.1. Experimental Design

The model of a randomized complete block design with a 4 × 4 factorial arrangement (Montgomery, 2009) is:

$$Y_{ijk} = \mu + \alpha_i + \beta_j + \gamma_k + (\alpha\beta)_{ij} + \varepsilon_{ijk}$$

Definitions of terms are as follows:

Mean Model Components:

μ the overall mean

Main Effect Model Components:

α_i the main effect of being in level *i* of factor A (vacuum pressure)

β_j the main effect of being in level *j* of factor B (storage time)

γ_k the main effect of being in level *k* of blocking factor C (powder type)

Two-way Interaction Model Components:

$(\alpha\beta)_{ij}$ the effect of being in:

level *i* of factor A(vacuum pressure) and level *j* of factor B (storage time)

Error Components:

ε_{ijk} the unexplained part of the score

i = 1, 2, 3, 4 (four levels of applied vacuum pressure)

1 = 1 bar (roughly equal to 99% vacuum pressure in the bag)

2 = 0.7 bar (roughly equal to 50% vacuum pressure in the bag)

3 = 0.4 bar (roughly equal to25% vacuum pressure in the bag)

4 = 0 bar (No vacuum in the bag)

It is important to notice that effect of vacuum pressure was called "Packaging conditions" and abbreviated to "Pack-co" in most tables and graphs, as in fact it was a combination of the effects of vacuum pressure and packaging material including the following levels:

1 = Kraft (0% vacuum pressure + Kraft paper)

2 = 99vacpet (99% vacuum pressure + multiwall PET bags)

3 = 50vacpet (50% vacuum pressure + multiwall PET bags)

4 = 25vacpet (25% vacuum pressure + multiwall PET bags)

5 = 0 vacpet (No vacuum pressure + multiwall PET bags)

j = 1, 2, 3, 4 (four levels of storage time)

1 = time 0 (right upon receiving and before vacuum packaging)

2 = after 3 month storage

3 = after 6 month storage

4 = after 12 month storage

k = 1, 2, 3, 4, 5, 6 (six levels of powder type)

1 = WMP

2 = BMP

3 = NFDM

4 = SWP

5 = WPC80

6 = MPI

4.2.2. Statistical Analysis

The statistical analyses of all the quality attributes examined including: particle size, particle density, bulk density, tapped density, angle of repose, compressibility, moisture content, solubility, and color values L-, a-, and b. for 3, 6, and 12 month storage time were conducted using the GLM command in Minitab (v.16.1, Minitab Inc., State College, Pennsylvania). Powder type was a random effect. Storage time, vacuum pressure and the interaction between them were fixed effects. All statistical tests were performed at a significance level of $\alpha = 0.01$. To compare the changes in the quality attributes of the powders over the storage time, with their initial (upon receiving and before repackaging) quality, for each quality attribute of each powder, the differences between the initial value and the values collected at 3, 6, 12 month storage time were used for the statistical analyses. Tukey's method was used to compare treatment means. In addition histograms of residuals, normal plot of residuals, plots of residuals versus fits, and plots of residuals versus order were used to test model adequacy.

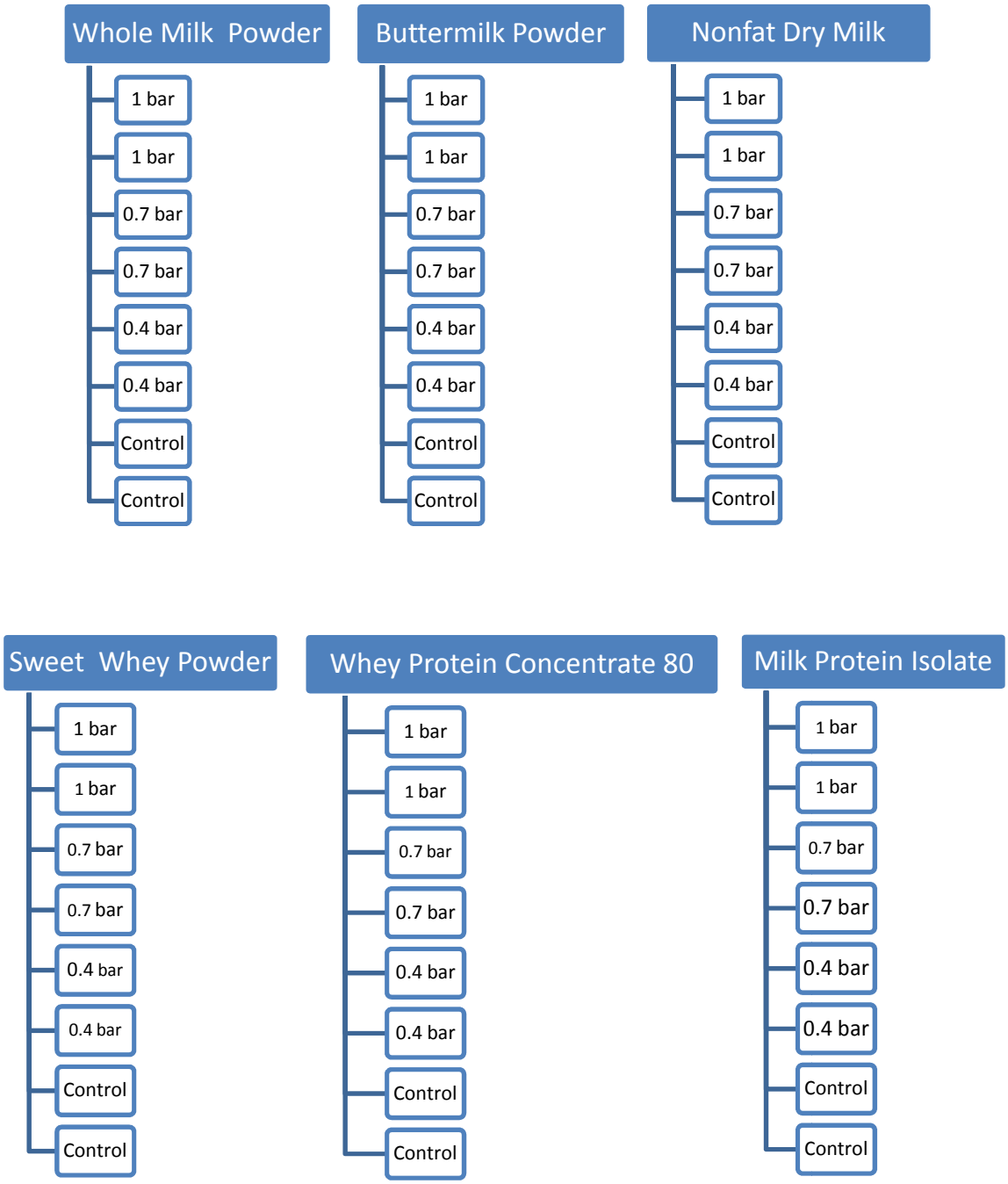

Figure 25: Schematic of powder vacuum packaging; packaging was done according the design above, in triplicate in order to produce three sets of samples for the three sampling times (3, 6, and 12 month storage period)

4.3. Chemical Composition of powders

4.3.1. Determination of Nitrogen and Protein

The levels of total nitrogen (TN) were determined via Kjeldahl method (AOAC, 1995). All measurements were carried out in duplicate. The total protein (TP) was calculated as TN × 6.38.

4.3.2. Determination of Moisture, Fat Content, Ash, and Lactose

Moisture was determined by drying each sample for 5 h in a vacuum oven at 100ºC (American Dairy Products Institute, 1990). Fat content was determined by the Mojonnier ether extraction method as described by Marshall (1992).

Ash content was determined by ignition for 16 h at 550ºC in an electric furnace (AOAC, 1995). All measurements were carried out in duplicate.

Content of lactose + lactic acid was calculated by difference [total solid – (total protein + fat + ash)] as proposed by Guzman- Gonzalez et al. (1999)

4.4. Sampling method

At 3, 6, and 12 month after storage, each bag was cut and the powder was transferred to a new resealable Mylar bag (Polyester fill laminated to aluminum foil) (Impak Corporation, CA). In fact, the Mylar bag was used as a temporary packaging for the powder during the testing period. Then the powder in the Mylar bag was hand shaken for 10 times, to reaerate and disturb any lumps formed under compaction over the storage period. This procedure was repeated consistently for all powders at each sampling time.

4.5. Physical Properties of powders

4.5.1. Bulk Density

4.5.1.1. Loose/ poured bulk density

Bulk density of a powder is defined as the weight of a powder divided by the volume it occupies, normally expressed as g/ml or kg/l. Bulk density of the samples was measured according to IDF standard 134A:1995 method (IDF.Standard.134A, 1995) with some modifications. The principle of which is to weigh a known volume of a powder and calculate the density simply by dividing the measured weight per the known volume. The tests were performed according to the following procedure:

A 100 cm^3 glass graduated cylinder was placed on an analytical scale (with a sensitivity of 0.1 mg) and the scale was tared with the cylinder on it. Then the cylinder was filled with powder up to 100 cm^3 using a metal spoon without shaking or tapping the cylinder. As the scale had been tarred with the empty cylinder on it beforehand, it showed the weight of the powder (Svarovsky, 1987). The loose/ poured bulk density was expressed as:

$$loose\ (poured) bulk\ density = \frac{Wx}{100}$$

$Wx = weight\ of\ powder\ in\ g$

$100 = volume\ of\ cylinder\ in\ cm^3$

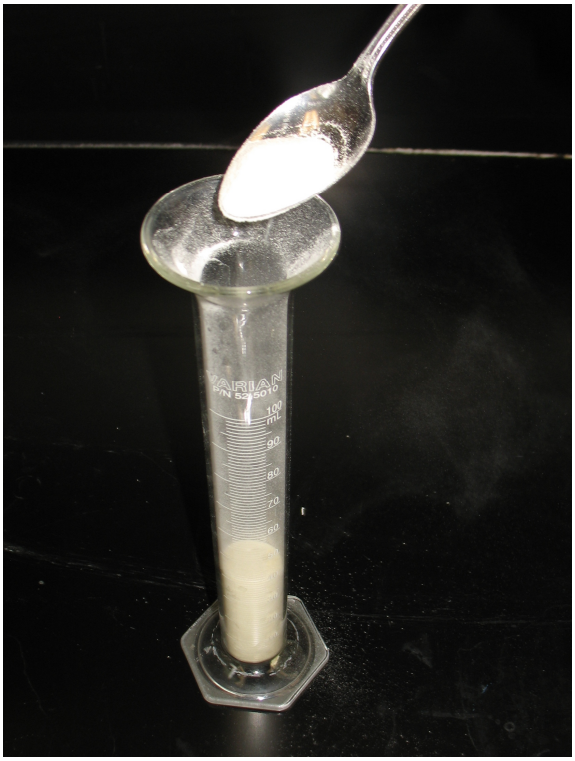

Figure 26: Pouring nonfat dry milk into a 100 ml cylinder to measure bulk density

The results were calculated to 2 decimal places and reported in g/cm^3. Measurements were conducted in duplicate.

4.5.2. Tapped Density

Tapped density of a powder is defined as the weight of a powder divided by the volume it occupies after tapped for certain times, normally expressed as g/ml or kg/l. Tapped density of the samples was measured according to IDF standard 134A:1995 method (IDF.Standard.134A, 1995) with some modifications. The principle of which is to weigh a known volume of a powder, tap it for 150 times on a Varion tapped density tester (Varion Inc., CA, USA) and calculate the density simply by dividing the measured weight per the tapped volume. The tests were performed according to the following procedure:

A Varion 100 cm^3 tapped density specific glass graduated cylinder was placed on an analytical scale (with a sensitivity of 0.1 mg) and the scale was tarred with the cylinder on it. Then the cylinder was filled with powder up to 100 cm^3 using a metal spoon without shaking or tapping the cylinder. As the scale had been tarred with the empty cylinder on it beforehand, it shows the weight of the powder. Then the cylinder is placed on the base of a Varion tapped density tester (Varion, Inc., NC, USA), tapped for 150 times. The tapped density is expressed as:

$$\text{Tapped density} = \frac{\text{Wx}}{\text{Tapped volume}}$$

$Wx = weight\ of\ powder\ in\ g$

$Tapped\ volume = volume\ of\ powder\ after\ tapped\ for\ 150\ times\ in\ ml$

The results were calculated to 2 decimal places and reported in g/ml. Measurements were conducted in duplicate.

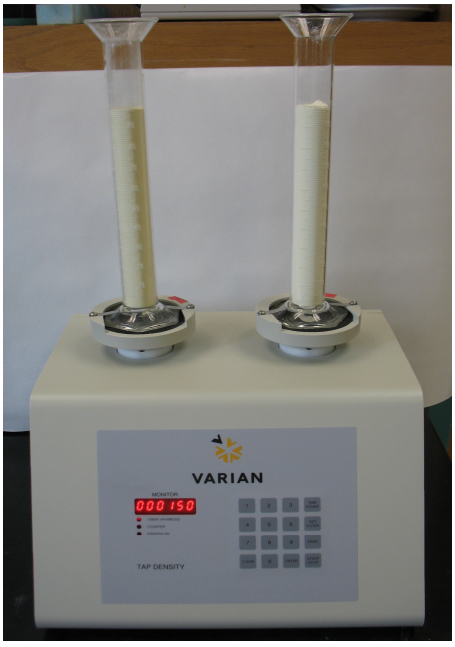

Figure 27: Varion tapped density tester

4.5.3. Particle Density

Particle density of the samples was measured according to the air pycnometer method of GEA Niro analytical methods (GEA.Niro.method.A.11.a) with some modifications. The principle of the method is to determine the true volume of a sample (the volume in g/cm^3 enclosed by its outer surface and excluding its open pores) by measuring the pressure change of helium in a calibrated volume.

An AccuPyc 1330 air pycnometer (Micromeritic Instrument Corporation, GA, USA), was used for particle density measurements. The pressure of the helium was adjusted to 2 bars on the gas flask. The parameters were checked by pressing the blue button and button No. 2 on the keypad. Then the 'Enter' key was pressed. The parameters were set up as follows: (Number of purges: 5, Purge fill pressure: 19.5 psig, Number of runs: 5, Run fill pressure: 19.5 psig, Equilibration rate: 0.050 psig/min, Use run precision: No). Then the 'Save' key was pressed to store the information. The display should show 'Reload'. An amount of powder (usually equal to 2/3 of the sample cup's volume) was weighed into the sample cup using an analytical balance, capable of weighing to 0.1 mg and the excess powder on the sides of the cup was removed using a brush. The chamber cap was removed by turning it counter clockwise, then lifting up. The sample cup was inserted in the cell chamber and the chamber cap was put on again. The blue button and button No. 4 were pressed. The sample identification was typed, followed by 'Enter' and the sample weight followed by 'Enter'. To start the analysis 'Enter' was pressed. When the analysis stopped (after approx. 10-12 min.) the results were recorded in 4 decimal places and reported in g/cm^3. Measurements were conducted in duplicate.

Figure 28: Accupyc 1340 gas pycnometer

4.5.4. Particle Size Diameter Distribution Analysis

Particle size diameter distribution of powders was determined using Coulter LS 230 with Dry Powder Module (Beckman Coulter, Brea, CA). The instrument was allowed to adjust for electrical offsets and align the laser prior to measuring background. Background was measured for 60 sec. Sample loading was measured for 60 seconds. Obscuration was held between 4% and 7% during the runtime by adjusting speed of the auger attached to the feed mechanism. Voltage measurements from the detector were converted to particle size diameter distributions by Beckman Coulter LS Software v.3.29 August 2003 (Brea, CA). Measurements were conducted in triplicate.

4.5.5. Angle of Repose

The angle of repose was tested as an indicator of powder flowability, using the Geldart MARK4 Angle of Repose Tester (Invented by Professor Derek Geldart, Powder Research Ltd, UK), according the procedure below:

4.5.5.1. Geldart MARK4 Angle of Repose Tester (Figure 31)

A representative sample of the powder was taken using a metal spoon. About 100 grams is the optimum quantity, and the weight ± 1g was recorded, but samples as small as 25g may be used if only a small mass of powder is available. As several different powders were tested, the same mass of sample was used for each test. Using a small metal scoop, the powder was slowly poured on to the upper part of the large converging chute taking about 20 seconds for the entire sample. Whenever the sample showed signs of cohesiveness, such as sticking on the chute, or an inability to slide, the sample was recovered and the test was repeated with the vibratory motor switched on, once again taking about 20 seconds to pour the entire sample on to the chute.

The powder semi-cone should have a well-defined, sharp apex. Sometimes, if the pouring has been done too quickly or the powder has become excessively aerated the apex may be flattened making it difficult accurately to read the height "h", of the semi-cone. Whenever this occurred, the test was discarded. [This is more likely to occur with very cohesive powders]

After each test, the powder was recovered by pouring it from the base plate into a metal container, taking care to remove all the powder, including any fine dust, using a soft brush.

The height h of the semi-cone was recorded. The divisions on the scales are 2 mm apart so it is possible to take readings to within 1 mm by visual interpolation. The radius "r" of the base of the powder semi-cone was taken at 3 to 5 positions, and the average value of "r" was used.

Figure 29: Semi-cone of buttermilk powder with a well-defined and sharp apex

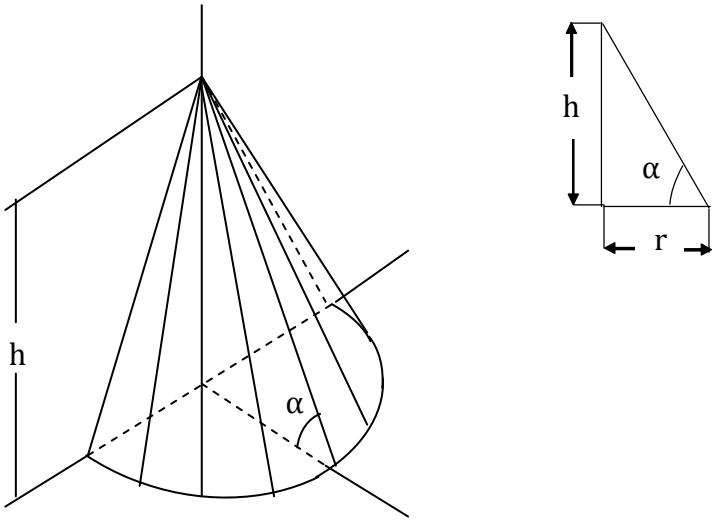

Figure 30: Schematic of Angle of Repose (α)

Angle of repose was then calculated from equation below: (Measurements were conducted in duplicate)

$$\mathbf{AOR\alpha = \tan^{-1}(h/r)}$$

The powder sample was recovered as described earlier in this section and the test was repeated as many times as required, until the standard deviation was acceptable.

The additional items needed for the test were: a polished, smooth, stainless steel container (about 1 Liter capacity), a small, polished smooth stainless steel scoop (plastic scoop should not be used because it may cause electrostatic charging of the powder), a small soft paint brush

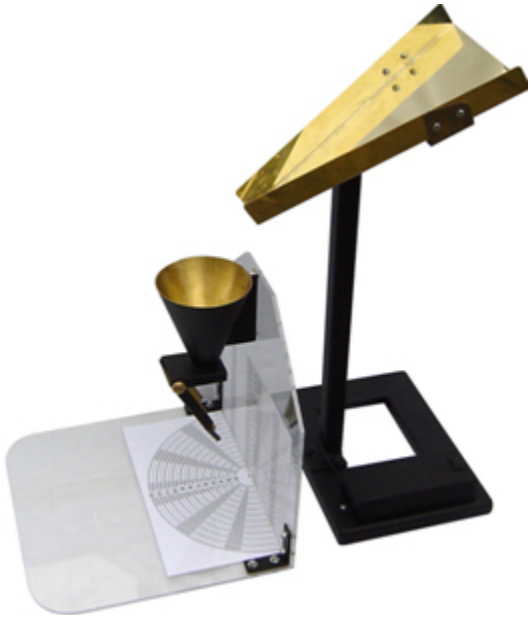

Figure 31: Geldart Mark 4 Angle of Repose Tester

4.5.6. Compressibility

Compressibility was tested using a Texture Analyzer – TA-XT2 (Stable Micro Systems) with a piston in a cylinder (cylindrical acrylic cup 55 × 70 mm and a 45 mm diameter disk) and a back extrusion A/BE assembly, according to the method described by Eduardo and Lannes, (2007). The tested powder was poured into the cylinder without any tapping. The excess powder was scraped off the top of the cylinder, using a metal

knife. Then the filled cylinder was placed on the base of the texture analyzer and the powder was compressed with a piston attached to the cross-head of the TA-XT2 Texture Analyzer. The analysis parameters were: Speed: 2 mm/s, Time: 5 s, Distance: 12 mm. A force-distance relationship during the compression test was recorded by the instrument. The probe height (distance between the bottoms of the piston disk to the top of the sample in the cylinder) was calibrated before running each test. The calibration parameters were: Return distance: 25 mm, and Contact force 5 (g).

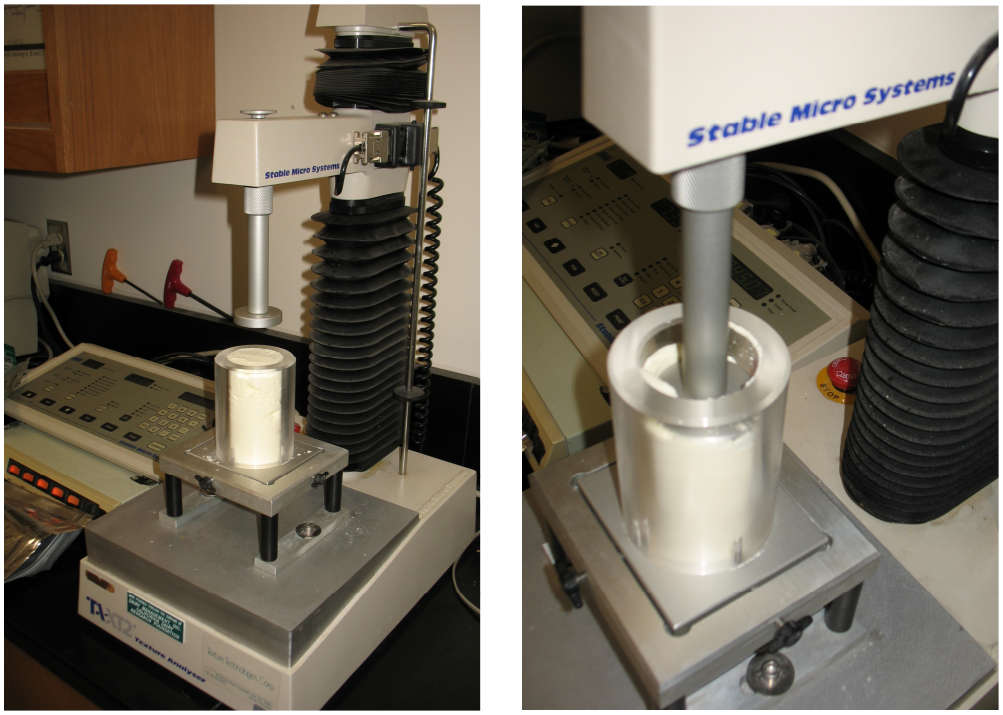

Figure 32: Texture analyzer – TA-XT2 with a piston in a cylinder and a back extrusion A/BE assembly

Keeping the compaction force and test speed constant, the distance each powder allowed the piston to travel into the sample cup, was recorded as an indicator of the compaction characteristics and compressibility of each powder and reported in mm distance. Measurements were conducted in duplicate.

The more compactable was the product, the longer was the distance traveled by the piston, and the less compactable, the shorter was that distance.

4.5.7. Color

The color of powders was measured using an Ultra Scan XE Spectrophotometer (Hunter lab, Reston, VA). The color scale "CIE L*a*b*" was used as a full color descriptor. The instrument was standardized for "RSIN" and the large area of view, first using the light trap, then the white standard tile. The sample clamp was lowered and the shelf of the reflectance sample shelf with light cover (HunterLab Part Number B02-1005-172) was installed at the reflectance port. The sample powder was scooped up from the sample batch to fill the 50-mm glass cell (HunterLab Part Number 13-8573-20) to the top. The sample cup was tapped once on a hard surface to settle the loose powder and then the filled cell flush was placed against the reflectance port so that the powder will be read through the clear glass window of the cell. Then the sample cell was covered with the opaque cover. A single color reading of the powder was taken. The powder was dumped, refilled, and read two times from the same batch. Average of the two color readings for a single color measurement was reported, representing the color of the batch. Averaging multiple readings minimizes measurement variation associated with non-uniform samples.

Figure 33: Color measurement using Ultra scan XE spectrophotometer

4.5.8. Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) Scanning electron microscopy (SEM) was performed on samples mounted on double-sided adhesive tape and attached to SEM aluminum stubs. Excess particles were removed by gently shaking the stub. Samples were then coated with gold in a Desk V HP series sputter coater (Denton Vacuum, LLC, Moorestown, NJ). The samples were examined with a FEI Quanta 200 SEM instrument (FEI Company, Hillsboro, OR) operated at spot size 4.5 and 10 kv accelerating voltage.

4.6. Solubility Analysis

4.6.1. Insolubility Index

One-hundred ml of water (24 ˚C ± 0.2 ˚C) was poured into the mixing jar (Waring Commercial Blender [Model 34B197, 120 Volts AC, 50 Hz to 60 Hz, 7.0 Amps], Torrington, CT). An aliquot of MPC (5 g) was placed into the mixing jar. The analysis then proceeded according to insolubility index: GEA Niro analytical method A 3a (Niro,

2010). Three drops of Antifoam B Emulsion (Sigma-Aldrich, St. Louis, MO) were added, and mixing commenced at 3800 rpm for 90 sec. The solution was allowed to sit for 15 min. After 15 min. elapsed, the solution was stirred with a thin spatula and transferred to two 50 ml centrifuge tubes. Centrifugation took place at 910 rpm for 5 min. A pipette was used to dispose of all sediment-free liquid more than 5 ml above the sediment layer. The centrifuge tubes were then filled with DI water to the 50 ml mark, the sediment was dispersed with a thin spatula, and centrifugation again took place at 910 rpm for 5 min. The volume of remaining sediment was reported in ml. Measurements were conducted in duplicate.

4.7. Storage Space Analysis

Storage space analysis was performed by comparing three geometric models: one for atmospheric packaged (model 1) and two for vacuum packaged (models 2 and 3). Model1 represents the dimensions (length, width, and height) of an original 25 kg atmospheric packaged bag of NFDM. Model 2 represents a 25 kg vacuum packaged bag of NFDM with a width and length as the original bag but a shorter height, due to the effect of vacuum packaging. To achieve the highest pallet efficiency (for a 48 × 40 in pallet) model 3 was designed to represent a 25 kg vacuum packaged bag of NFDM with "a width = one third of the length of the pallet" and "a length = half of the width of the pallet" (so that the dimensions suit a 3 × 3 pallet pattern).The height of model 2 (H_2) and model 3 (H_3) were calculated using the known weight of the bags (25 kg), the density of the vacuum packaged powder, and the known dimensions of the bags, as outlined in

Figure 34 The density of vacuum packaged powders was measured by submerging a vacuum packaged bag of NFDM in water, displacement of water and correcting for the weight and volume of an empty bag. The difference between the volume of models 1 and 2 was reported as the saved space per bag. As there are 45 bags on each pallet of dairy powders commonly, the saved space was multiplied by 45 to get the saved space per pallet, as outlined in Figure 34.The CAPE PACK v2.09 software was also used to measure the efficiency of using the storage space.

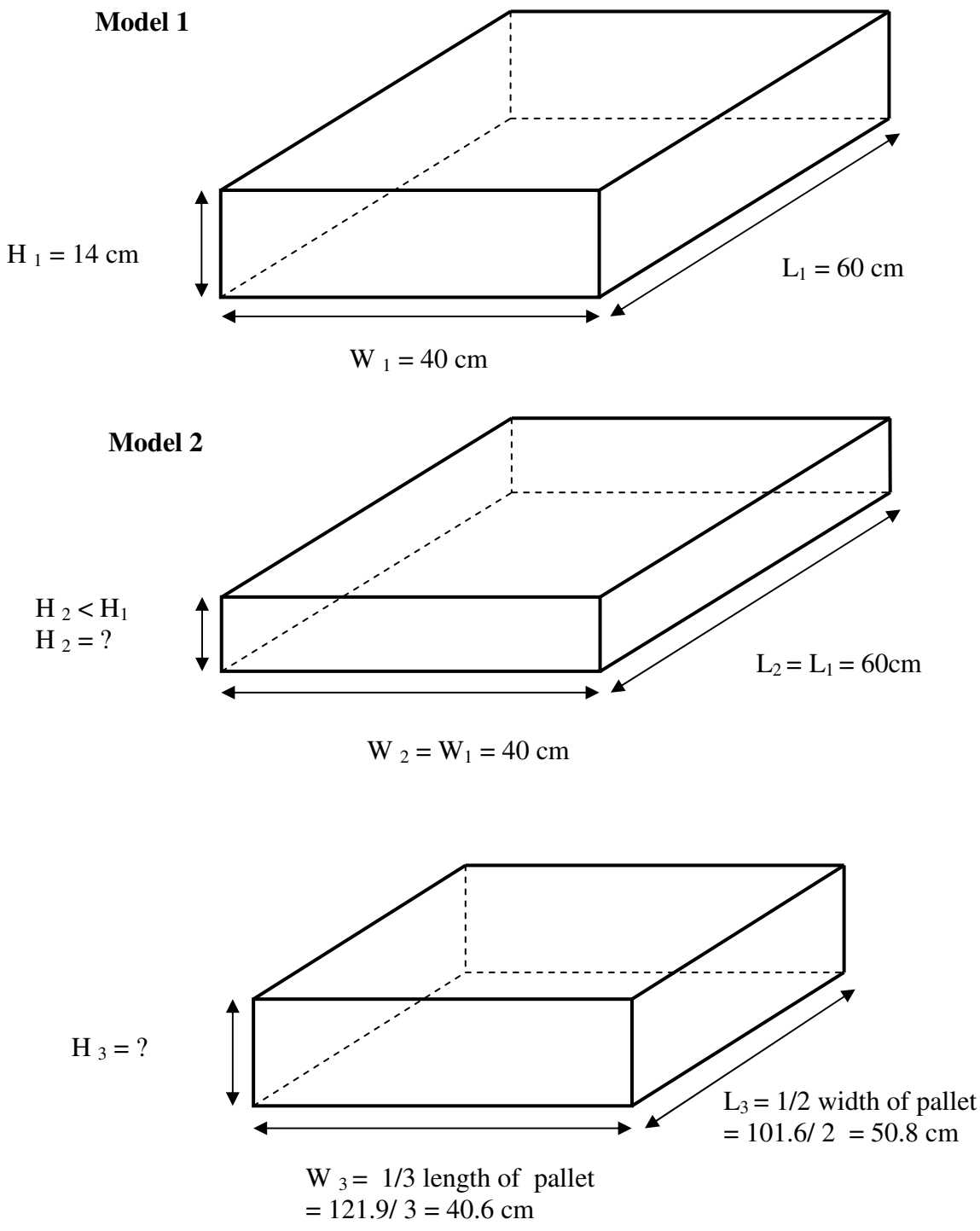

Figure 34: Schematic models of a 25 kg bag of atmospheric packaged (model 1) versus vacuum packaged NFDM (models: 2 and 3), model 2: saving space in height; model 3: saving space in length

5. RESULTS AND DISCUSSION

5.1. Compositional properties of powders

The data collected according to the method outlined in sections 4.3.1 and 4.3.2, pertaining to total protein (TP), fat content, and lactose, is shown in Appendix A.

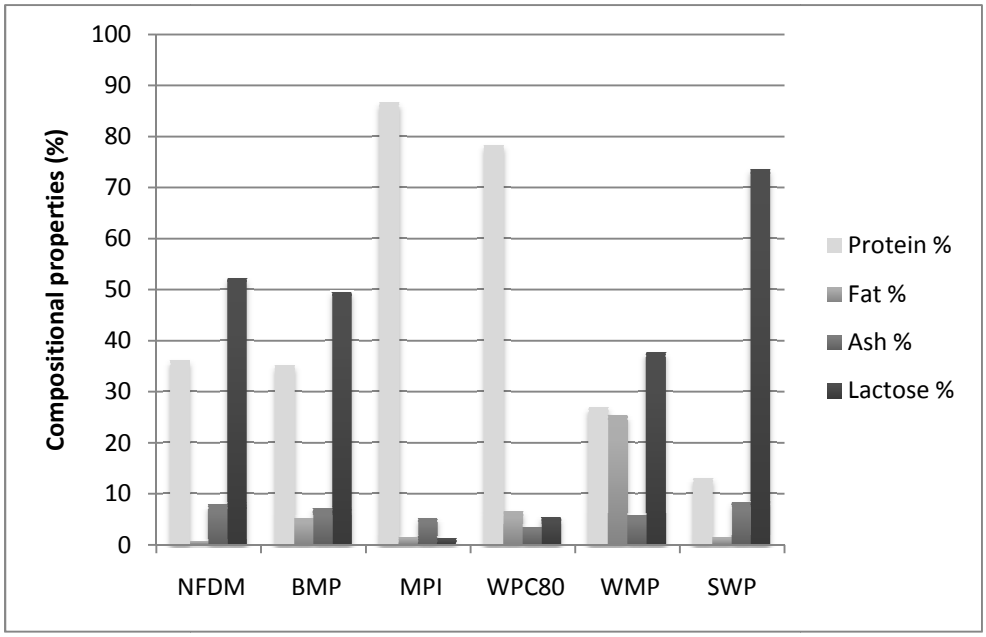

Figure 35: Compositional properties of powders (%) (protein, fat, ash, and lactose)

Protein content

MPI > WPC80 > NFDM > BMP > WMP > SWP

Protein contents of the powders were consistent with each other and conforms to the typical range offered by American Dairy Products Institute (ADPI). As expected; MPI had the highest and SWP has the lowest protein content.

Fat content

WMP > WPC80 > BMP > MPI > SWP > NFDM

Fat contents of the powders were consistent with each other and conforms to the typical range offered by American Dairy Products Institute (ADPI). As expected; WMP has the highest and NFDM has the lowest fat content

Ash content

SWP > NFDM > BMP > WMP > MPI > WPC80

Ash contents of the powders were consistent with each other and conforms to the typical range offered by American Dairy Products Institute (ADPI). As expected; SWP has the highest and WPC80 has the lowest ash content

Lactose

SWP > NFDM > BMP > WMP > WPC80 > MPI

Lactose contents of the powders were consistent with each other and conforms to the typical range offered by American Dairy Products Institute (ADPI). As expected; SWP has the highest and MPI has the lowest lactose content

5.2. Effect of vacuum packaging and storage time on particle density

The data collected according to the method outlined in section 4.5.3, pertaining to particle density, is shown in Appendix B. The statistical analysis of particle density on Accupyc 1340 pycnometer was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there were no statistically significant differences in mean particle density due to the effect of vacuum pressure ($p = 0.926$). The interaction between storage time and vacuum pressure was not statistically significant ($p = 0.679$). However, there was a statistically significant difference in particle density due to the effects of storage time ($p = 0.000 < 0.01$) and powder type ($p = 0.000 < 0.01$). The mean for each of the

powders at different levels of treatment (vacuum pressure and storage time) are displayed in Table 5.

Table 5: Particle density (g/ ml) of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	1.2879	1.2455	1.1472	1.2402	1.4594	1.2522
3	99vacpet	1.3073 ± 0.00	1.2585 ± 0.00	1.1606 ± 0.00	1.2466 ± 0.00	1.4727 ± 0.00	1.2610 ± 0.01
3	50vacpet	1.3067 ± 0.00	1.2580 ± 0.00	1.1623 ± 0.00	1.2479 ± 0.00	1.4689 ± 0.00	1.2604 ± 0.00
3	25vacpet	1.3046 ± 0.00	1.2550 ± 0.00	1.1602 ± 0.00	1.2453 ± 0.00	1.4750 ± 0.00	1.2669 ± 0.00
3	0vacpet	1.3046 ± 0.00	1.2550 ± 0.00	1.1602 ± 0.00	1.2453 ± 0.00	1.4736 ± 0.00	1.2669 ± 0.00
6	99vacpet	1.3116 ± 0.00	1.2525 ± 0.00	1.1728 ± 0.00	1.2473 ± 0.00	1.4738 ± 0.00	1.2615 ± 0.00
6	50vacpet	1.3114 ± 0.00	1.2508 ± 0.00	1.1735 ± 0.01	1.2479 ± 0.00	1.4732 ± 0.00	1.2764 ± 0.00
6	25vacpet	1.3124 ± 0.01	1.2551 ± 0.00	1.1714 ± 0.00	1.2480 ± 0.00	1.4730 ± 0.00	1.2714 ± 0.00
6	0vacpet	1.3101 ± 0.00	1.2533 ± 0.00	1.1747 ± 0.00	1.2473 ± 0.00	1.4718 ± 0.00	1.2723 ± 0.00
12	99vacpet	1.3206 ± 0.01	1.2743 ± 0.01	1.1721 ± 0.00	1.2710 ± 0.00	1.4746 ± 0.00	1.2826 ± 0.00
12	50vacpet	1.3167 ± 0.00	1.2722 ± 0.01	1.1764 ± 0.00	1.2667 ± 0.00	1.4722 ± 0.00	1.2809 ± 0.00
12	25vacpet	1.3127 ± 0.00	1.2768 ± 0.01	1.1710 ± 0.01	1.2675 ± 0.00	1.4718 ± 0.00	1.2793 ± 0.00
12	0vacpet	1.3140 ± 0.00	1.2745 ± 0.01	1.1704 ± 0.00	1.2640 ± 0.00	1.4718 ± 0.00	1.2790 ± 0.00

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

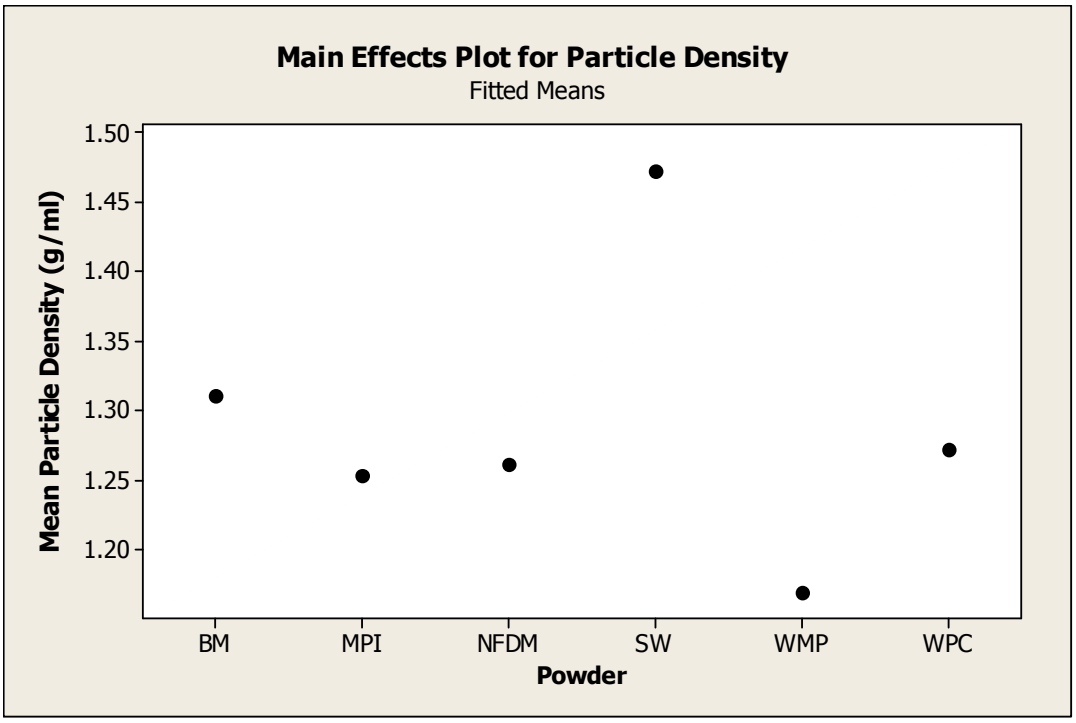

Figure 36: Main effects plot for particle density versus powder type

Figure 36 shows the significant effect of powder type on particle density. The differences observed can be related to a combination of many different factors such as variations in processing specifications, compositional properties, and particle structure of different powders. For example, it is known that:

- Rotary wheel atomizers tend to entrap more air than pressure nozzle atomizers and produce powders with lower particle densities (Robertson, 2006).
- High protein content tends to reduce particle density because more air is whipped into the feed during atomization. This explains the higher particle density observed in a low protein powder such as SWP (Pisecky, 1986). However, this theory fails to explain the low particle density observed in milk protein isolate with high protein content.

- Increasing total solids content reduces the quantity of air entrapped in powder particles, and thus increases particle density. As the solids content increases, viscosity increases exponentially and it becomes more difficult to whip air into a more viscous liquid (Nielsen et al, 1982). This might be a possible explanation for the low particle density observed in milk protein isolate.
- Powder particles with higher number of vacuoles have lower particle densities. Vacuoles in the powder are formed by the expansion of entrapped air in the droplets during spray drying (Verhey, 1972).

As the significant effect of powder type on particle density is highly dependent on the manufacturing specifications of each powder, in order to explain it, having the knowledge of the manufacturing details of each powder is required.

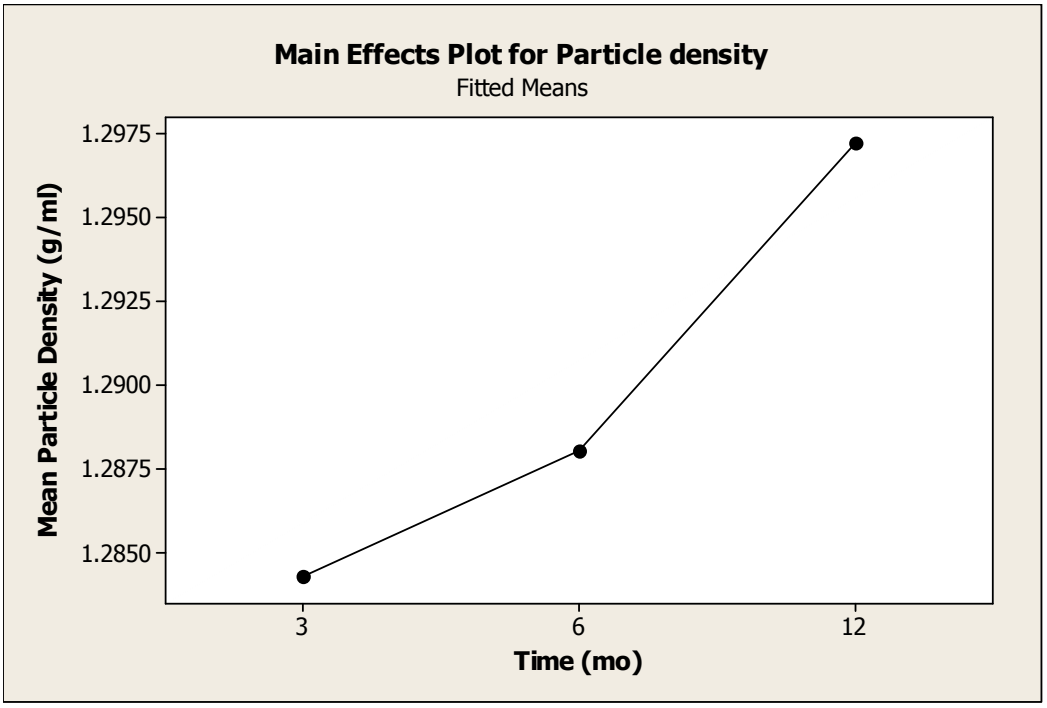

Figure 37: Main effects plot for particle density versus storage time

Figure 37 shows a significant effect of storage time on particle density with an upward trend over the storage time. The increase in particle density over the storage time may be related to time consolidation.

Particle density influences bulk density, flowability, compressibility, solubility of the powders.

Time consolidation

When considering the behavior of a powder, an important factor that must not be overlooked is the time the material is likely to be at rest and under load. In general, there is a tendency for the strength of a powder to increase over time if allowed to consolidate. There may be several reasons for this:

- A de-aeration and rearrangement on the particle level over time; here, the material settles into a more compact and stronger configuration. There may be some problem due to an increase in moisture content of the bulk solid, for example from moist warm air, resulting in condensation (McGlinchey, 2009).
- Or there may be some physical and chemical reactions taking place, forming solid bridges between individual particles, which can result in a strong material for which it is very difficult to initiate flow (Teunou and Fitzpatrick, 1999).

Powder consolidation over time may cause an increase in bulk density, which leads to the powder particles being pushed closer together. This results in increased van der Waals interaction leading to greater cohesion, and forming powder particles with higher densities (Bhandari and Hartel, 2005). Physical and chemical changes often

require time, such as the migration of liquids or the crystallization of sugars, which lead to increased cohesion or even caking over time.

5.3. Effect of vacuum packaging and storage time on particle size

The data collected according to the method outlined in section 4.5.4., pertaining to particle size, is shown in Appendix C. The statistical analysis of the mean particle size was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there was no statistically significant difference in mean particle size due to the effect of vacuum pressure ($p = 0.\ 956$). The interaction between storage time and vacuum pressure was not statistically significant ($p = 0.\ 995$). However, there were statistically significant differences in particle size due to the effects of storage time ($p = 0.000 < 0.01$) and powder type ($p = 0.000 < 0.01$). The mean particle size for each of the powders at different levels of treatment (vacuum pressure and storage time) is displayed in Table 6.

Table 6: Mean particle size (μm) of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	69.82	80.69	127.85	108	224.10	87.94
3	99vacpet	71.79 ± 0.69	82.43 ± 0.08	138.30 ± 2.12	110.34 ± 0.37	225.50 ± 1.27	86.85 ± 0.31
3	50vacpet	70.69 ± 0.16	83.19 ± 0.79	139.85 ± 1.34	108.92 ± 0.89	226.53 ± 0.74	88.48 ± 0.91
3	25vacpet	70.93 ± 1.10	83.28 ± 0.35	135.45 ± 1.06	109.55 ± 1.20	226.48 ± 0.11	90.34 ± 0.97
3	0vacpet	70.93 ± 0.25	83.73 ± 0.78	137.10 ± 0.42	111.14 ± 0.37	226.08 ± 0.25	88.23 ± 0.54
6	99vacpet	76.59 ± 0.03	85.59 ± 0.94	140.20 ± 2.12	105.94 ± 0.27	227.65 ± 1.13	91.72 ± 1.04
6	50vacpet	76.60 ± 0.94	85.72 ± 0.92	139.33 ± 0.32	106.83 ± 1.02	225.85 ± 1.06	92.70 ± 0.94
6	25vacpet	77.55 ± 0.63	85.17 ± 1.18	139.60 ± 1.98	107.45 ± 0.85	226.00 ± 1.27	93.38 ± 0.75
6	0vacpet	77.18 ± 0.31	84.90 ± 1.30	139.48 ± 1.81	107.08 ± 1.94	225.05 ± 0.14	93.40 ± 0.99
12	99vacpet	76.93 ± 0.70	85.03 ± 0.30	139.88 ± 2.23	113.50 ± 0.92	231.41 ± 0.40	95.69 ± 0.90
12	50vacpet	77.77 ± 0.83	84.41 ± 0.54	142.00 ± 3.61	111.93 ± 0.04	232.35 ± 1.63	96.07 ± 0.41
12	25vacpet	76.29 ± 1.58	85.87 ± 0.23	141.85 ± 0.42	111.03 ± 1.13	231.07 ± 0.33	97.20 ± 0.42
12	0vacpet	77.69 ± 1.83	85.03 ± 0.11	142.30 ± 1.48	110.83 ± 0.32	231.60 ± 0.99	97.70 ± 1.06

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

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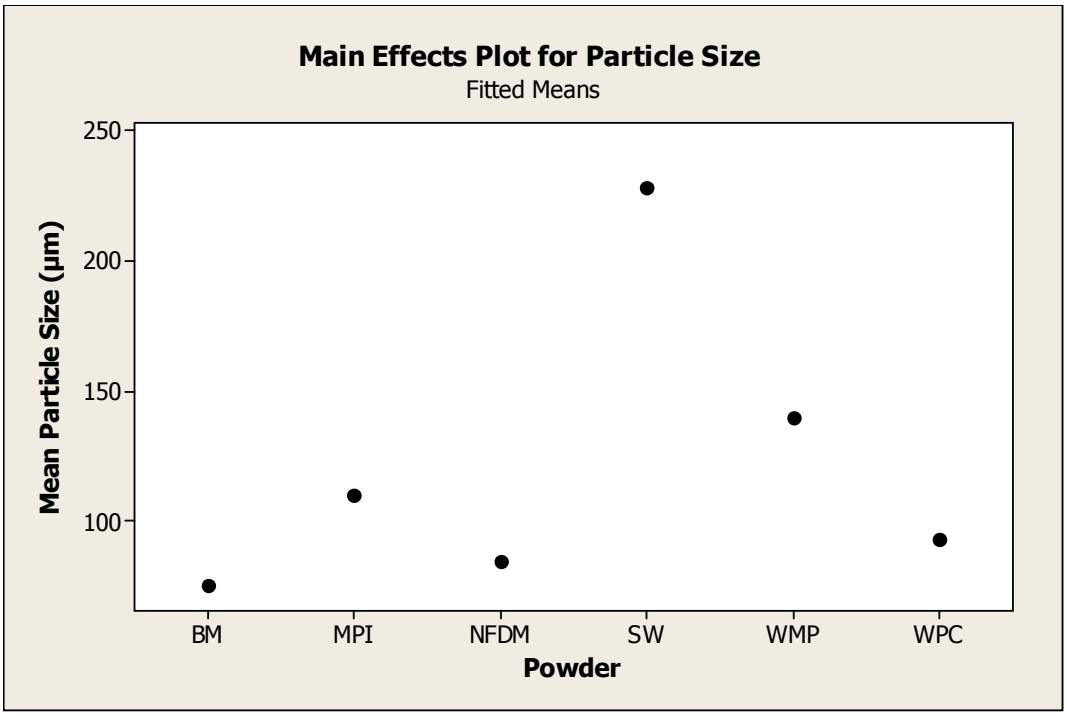

Figure 38: Main effects plot for particle size versus powder type

Figure 38 shows the significant effect of powder type on particle size. The differences observed may be mainly related to the differences in the compositional properties and processing specifications of the powders. Particle size can be influenced by the feed characteristics, processing conditions and the type of equipment used in the drying process. For example, when the feed has higher total solids content, each droplet contains more solids and forms a larger particle, while a low concentrate viscosity reduces particle size. Atomization parameters also influence shape and size distribution of powder particles. Spray-drying using centrifugal atomization gives a larger particle than pressure nozzle atomization (Singh and Newstead, 1992). However, as all the powders used were commercial powders, the type of nozzles used is unknown.

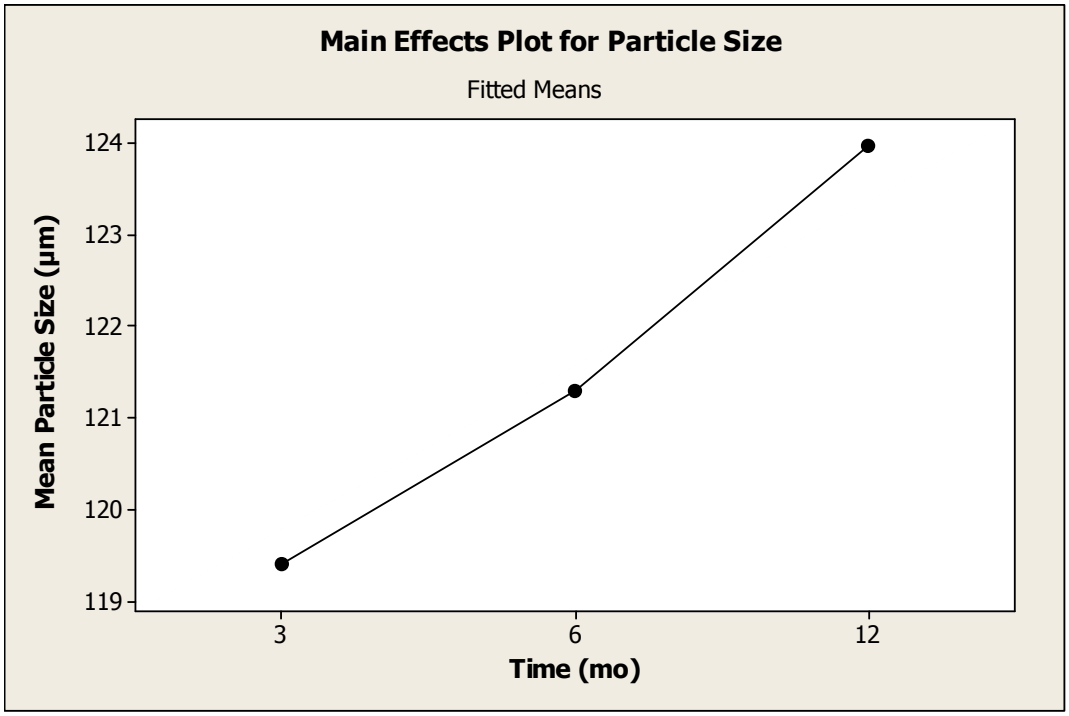

Figure 39: Main effects plot for particle size versus storage time (month)

Figure 39 shows the significant effect of storage time on particle size with an upward trend over the storage time. The increase in particle size over the storage time might have been caused due to time consolidation (as defined in section 5.2), which leads to the powder particles being pushed closer together. This results in increased van der Waals interaction leading to greater cohesion among particles (Bhandari and Hartel, 2005) and forming larger powder particles.

Particle size influences density, compressibility, flowability , solubility of the powders.

5.4. Effect of vacuum packaging and storage time on bulk density

The bulk density of the powders, as determined according to the IDF standard 134A:1995 method, outlined in section 4.5.1., is shown in Appendix D. The statistical

analysis of the mean bulk density was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there was no statistically significant difference in mean bulk density of the powders, due to the effect of vacuum pressure ($p = 0.\ 917$). The interaction between storage time and vacuum pressure was not statistically significant ($p = 0.762$). However, there were statistically significant differences in bulk density due to the effects of storage time ($p = 0.000 < 0.01$) and powder type ($p = 0.000 < 0.01$). The mean bulk density for each powder at different levels of treatment (vacuum pressure and storage time) is displayed in Table 7.

Table 7: Mean bulk density (g/ ml) of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements

.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	0.56	0.56	0.48	0.39	0.74	0.24
3	99vacpet	0.57 ± 0.00	0.57 ± 0.01	0.48 ± 0.01	0.41 ± 0.01	0.77 ± 0.00	0.25± 0.01
3	50vacpet	0.58 ± 0.00	0.57 ± 0.01	0.47 ± 0.01	0.40 ± 0.00	0.78 ± 0.01	0.25 ± 0.01
3	25vacpet	0.58 ± 0.00	0.57 ± 0.00	0.48 ± 0.01	0.40 ± 0.01	0.77 ± 0.00	0.25 ± 0.01
3	0vacpet	0.57 ± 0.00	0.58 ± 0.01	0.48 ± 0.00	0.39 ± 0.00	0.77 ± 0.00	0.24 ± 0.01
6	99vacpet	0.61 ± 0.00	0.59 ± 0.01	0.48 ± 0.00	0.41 ± 0.00	0.78 ± 0.01	0.28 ± 0.00
6	50vacpet	0.60 ± 0.00	0.58 ± 0.00	0.48 ± 0.01	0.41 ± 0.01	0.78 ± 0.01	0.28 ± 0.00
6	25vacpet	0.60 ± 0.01	0.59 ± 0.00	0.48 ± 0.01	0.41 ± 0.01	0.77 ± 0.00	0.28 ± 0.00
6	0vacpet	0.61 ± 0.00	0.59 ± 0.01	0.48 ± 0.00	0.41 ± 0.00	0.77 ± 0.01	0.28 ± 0.00
12	99vacpet	0.61 ± 0.01	0.59 ± 0.00	0.48 ± 0.01	0.41 ± 0.01	0.77 ± 0.00	0.29 ± 0.00
12	50vacpet	0.60 ± 0.01	0.59 ± 0.00	0.48 ± 0.01	0.41 ± 0.01	0.76 ± 0.00	0.28 ± 0.00
12	25vacpet	0.61 ± 0.00	0.58 ± 0.00	0.49 ± 0.00	0.41 ± 0.00	0.76 ± 0.00	0.28 ± 0.00
12	0vacpet	0.61 ± 0.00	0.58 ± 0.00	0.49 ± 0.01	0.41 ± 0.00	0.76 ± 0.00	0.28 ± 0.00

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

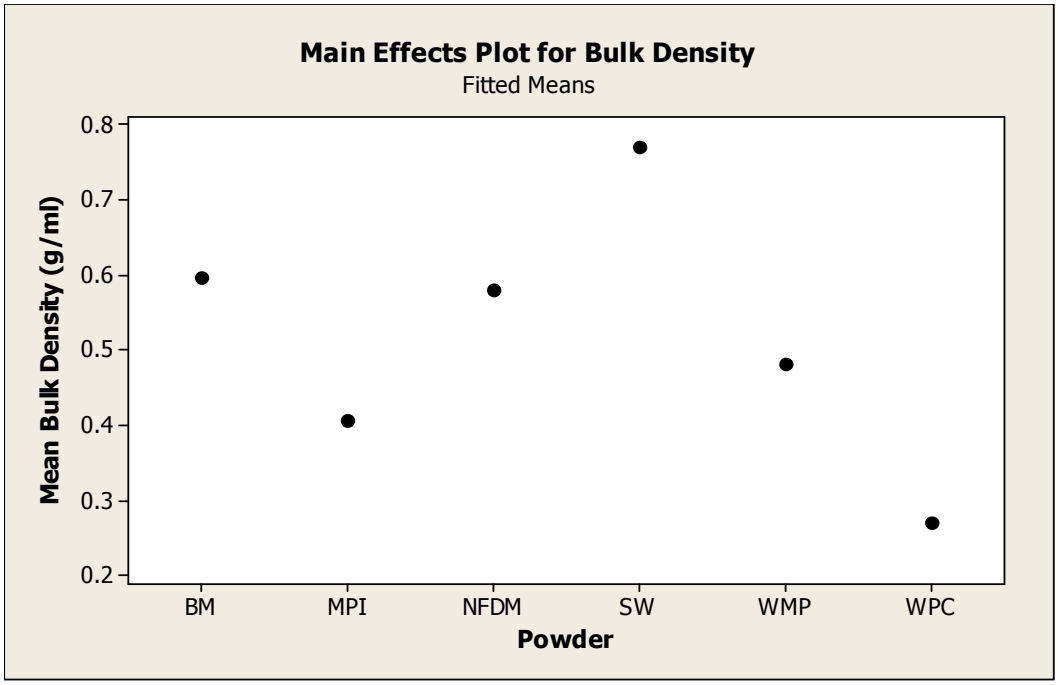

Figure 40: Main effects plot for bulk density versus powder type

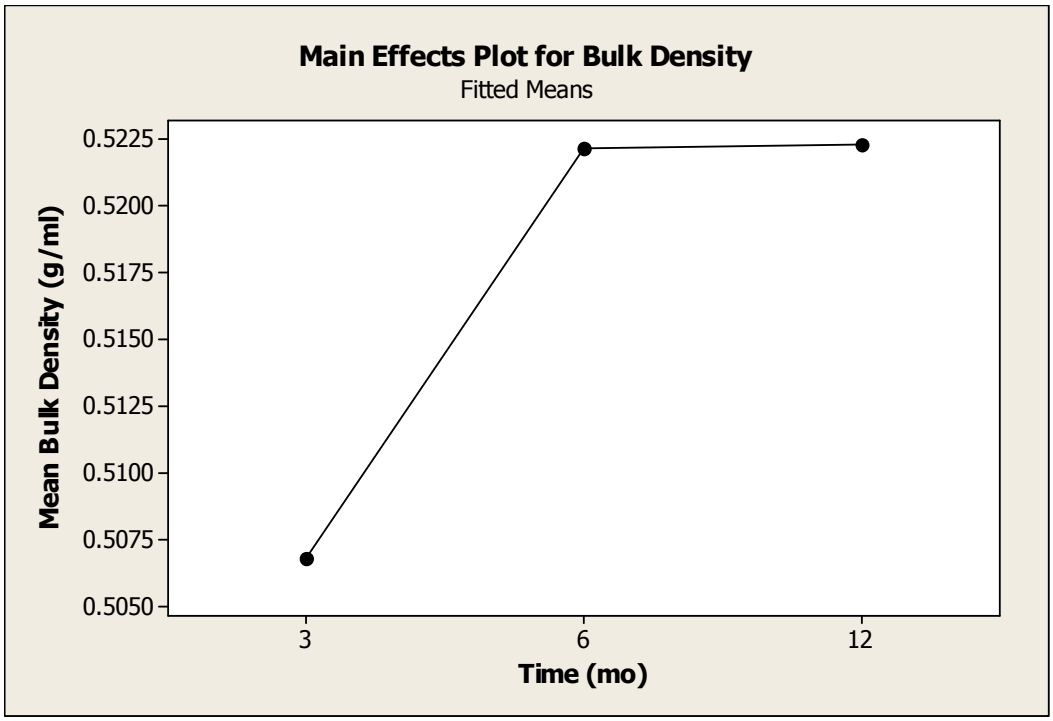

Figure 41: Main effects plot for bulk density versus storage time (month)

Figure 40 shows the significant effect of powder type on bulk density. Figure 41 shows the significant effect of storage time on particle size with an upward trend over the storage time. Since the bulk density of dairy powders depends on the combined effect of interrelated factors, such as the intensity of attractive interparticle forces, the particle size, and the number of contact points (Rumpf, 1961), it is clear that a change in any of the powder characteristics may result in a significant change in the powder bulk density. Therefore, the significant differences observed in the bulk density of the dairy powders studied in this project may be explained by the differences in the compositional properties and surface cohesion of the particles in different powders. The significant increase observed in the bulk density over the 12 month storage may be explained by the increased particle density and attractive interparticle forces caused by the time consolidation over the storage time. There seems to be an intricate relationship between the factors affecting dairy powder bulk density, as well as the surface activity and cohesion, which is well worth further inverstigation. Various milk fat fractions (surface free-fat, inner free-fat and encapsulated fat) can be extracted before and after storage, and analyzed for their fatty acid and triglyceride compositions to evaluate whether there was a release of fat onto the powder surface during storage and if this has caused any changes in the melting characteristics of the fat present on the powder surface. Moreover, the physical state of the lactose present in the powder can be studied before and after storage by X-ray diffractometer (XRD) to evaluate whether the lactose remained in amorphous state during storage.

Bulk density is important in determining the amount of powder that can be fitted into a bag or a bulk container.

5.5. Effect of vacuum packaging and storage time on tapped density

The tapped density of the powders, as determined according to the IDF standard 134A:1995 method, outlined in section 4.5.2., is shown in Appendix E. The statistical analysis of the mean tapped density, for 3, 6 and 12 month storage times were conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there was no statistically significant differences in mean tapped density of the powders, due to the effect of vacuum pressure ($p = 0.326$). The interaction between storage time and vacuum pressure was not statistically significant ($p = 0.642$). However, there were statistically significant differences in tapped density due to the effects of storage time ($p = 0.000 < 0.01$) and powder type ($p = 0.000 < 0.01$). The mean for each powder at different levels of treatment (vacuum pressure and storage time) are displayed Table 8.

Table 8: Mean tapped density (g/ ml) of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	0.76	0.77	0.60	0.60	0.95	0.38
3	99vacpet	0.78 ± 0.00	0.78 ± 0.00	0.61 ± 0.00	0.60 ± 0.01	0.96 ± 0.00	0.40 ± 0.00
3	50vacpet	0.78 ± 0.00	0.78 ± 0.01	0.60 ± 0.01	0.59 ± 0.01	0.96 ± 0.00	0.41 ± 0.01
3	25vacpet	0.78 ± 0.01	0.77 ± 0.00	0.61 ± 0.01	0.59 ± 0.00	0.97 ± 0.00	0.40 ± 0.00
3	0vacpet	0.77 ± 0.00	0.78 ± 0.00	0.61 ± 0.00	0.59 ± 0.00	0.96 ± 0.00	0.40 ± 0.01
6	99vacpet	0.78 ± 0.00	0.80 ± 0.00	0.62 ± 0.00	0.60 ± 0.00	0.97 ± 0.00	0.41 ± 0.01
6	50vacpet	0.82 ± 0.00	0.78 ± 0.00	0.62 ± 0.00	0.60 ± 0.01	0.97 ± 0.01	0.41 ± 0.00
6	25vacpet	0.81 ± 0.03	0.79 ± 0.01	0.61 ± 0.00	0.60 ± 0.00	0.96 ± 0.01	0.41 ± 0.00
6	0vacpet	0.82 ± 0.01	0.78 ± 0.00	0.61 ± 0.00	0.60 ± 0.00	0.96 ± 0.00	0.41 ± 0.00
12	99vacpet	0.83 ± 0.01	0.79 ± 0.00	0.62 ± 0.00	0.61 ± 0.00	0.96 ± 0.01	0.41 ± 0.00
12	50vacpet	0.83 ± 0.00	0.78 ± 0.00	0.63 ± 0.00	0.60 ± 0.00	0.96 ± 0.00	0.42 ± 0.00
12	25vacpet	0.83 ± 0.01	0.78 ± 0.00	0.62 ± 0.00	0.61 ± 0.00	0.96 ± 0.01	0.42 ± 0.00
12	0vacpet	0.82 ± 0.01	0.77 ± 0.01	0.62 ± 0.00	0.61 ± 0.00	0.95 ± 0.01	0.41 ± 0.00

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

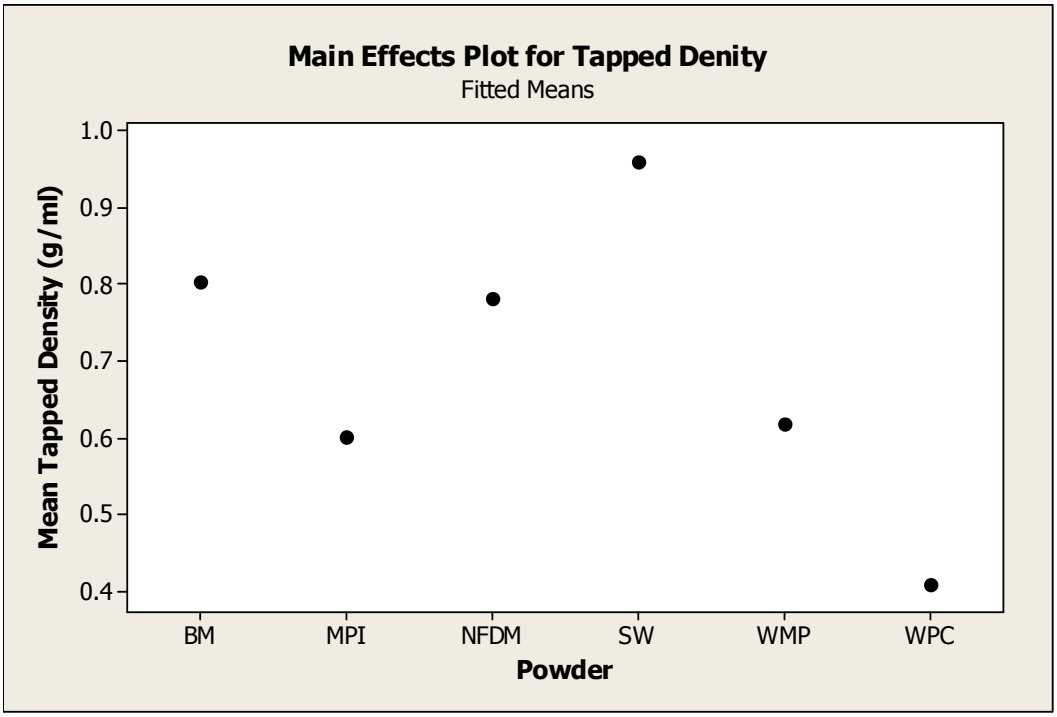

Figure 42: Main effects plot for tapped density versus powder type

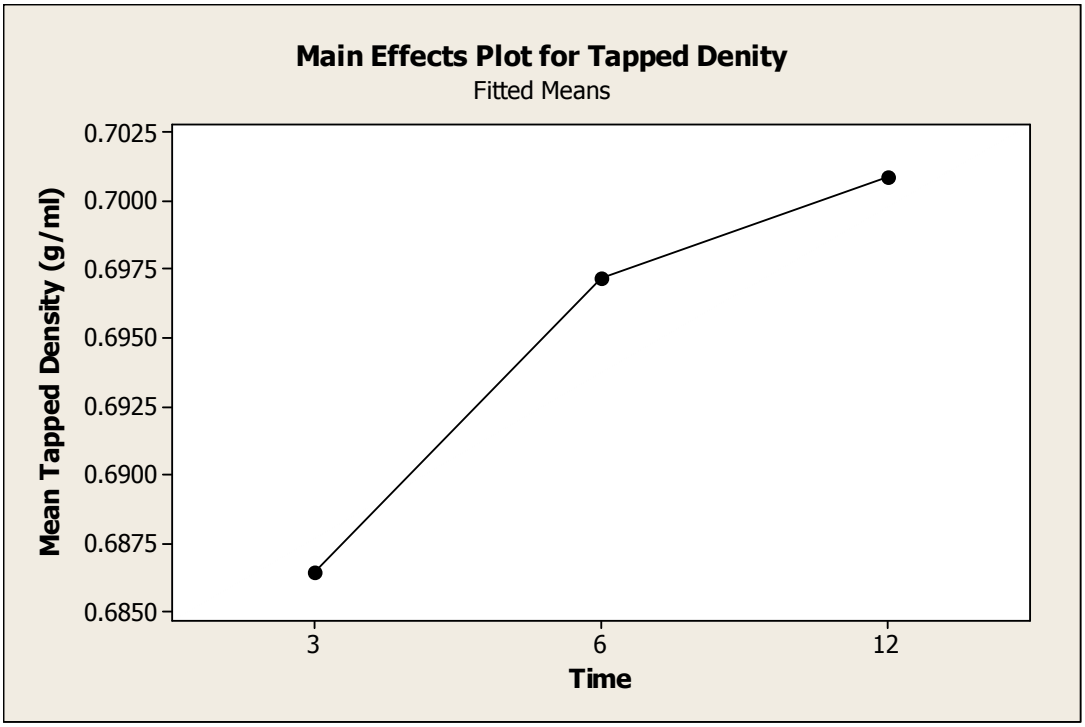

Figure 43: Main effects plot for tapped density versus storage time (month)

Figure 42 shows the significant effect of powder type on tapped density. The differences observed in compactibility can be explained by the differences in the compositional properties and interparticulate interactions of different powders.

Figure 43 shows the significant effect of storage time on tapped density with an upward trend over the storage time. The increase observed in tapped density over the storage time can be explained by the effect of time consolidation.

Tapped density is important in determining the amount of powder that can be fitted into a bag or a bulk container under tapping or vibration.

5.6. Effect of vacuum packaging and storage time on the angle of repose

The angle of repose (AOR) of the powders, as determined according to the method of Geldart MARK4 Angle of Repose (Geldart et al., 2006), outlined in section 4.5.5., is shown in Appendix F_1. The statistical analysis of the mean angle of repose was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there was no statistically significant difference in mean AOR of the powders, due to the effect of vacuum pressure ($p = 0.\ 980$). The interaction between storage time and vacuum pressure was not statistically significant ($p = 0.667$). However, there were statistically significant differences in AOR due to the effects of storage time ($p = 0.000 < 0.01$) and powder type ($p = 0.000 < 0.01$). The means for all the powders at different levels of treatment (vacuum pressure and storage time) are displayed in Table 9.

Table 9: Mean AOR (degree) of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	48.5	35.7	49.2	41.1	36.3	47.8
3	99vacpet	48.8 ± 0.3	32.3 ± 0.0	46.1 ± 0.8	41.0 ± 0.5	32.9 ± 0.4	44.9 ± 0.6
3	50vacpet	48.6 ± 0.0	33.7 ± 0.0	45.9 ± 2.4	40.5 ± 0.2	32.5 ± 1.6	44.3 ± 0.6
3	25vacpet	47.8 ± 0.3	35.4 ± 0.2	46.6 ± 0.2	40.8 ± 0.6	32.7 ± 0.1	44.6 ± 0.2
3	0vacpet	47.5 ± 0.7	35.4 ± 0.2	45.0 ± 0.5	41.6 ± 1.2	32.2 ± 1.4	44.5 ± 0.8
6	99vacpet	47.2 ± 0.4	33.9 ± 0.0	46.6 ± 0.1	41.6 ± 0.1	33.4 ± 0.6	44.9 ± 0.8
6	50vacpet	46.7 ± 0.9	34.3 ± 0.7	47.6 ± 1.3	41.4 ± 0.4	32.9 ± 0.1	46.5 ± 0.6
6	25vacpet	45.6 ± 0.5	35.1 ± 1.1	46.7 ± 0.1	40.5 ± 0.0	32.9 ± 2.1	45.0 ± 0.9
6	0vacpet	46.9 ± 0.8	35.0 ± 0.6	46.5 ± 0.0	41.2 ± 0.9	32.6 ± 0.6	46.1 ± 0.7
12	99vacpet	47.1 ± 0.5	30.8 ± 0.5	47.1 ± 0.5	39.0 ± 0.7	33.9 ± 0.7	44.3 ± 0.6
12	50vacpet	46.7 ± 0.3	30.3 ± 1.2	46.6 ± 0.3	39.0 ± 0.2	32.3 ± 0.0	44.0 ± 0.3
12	25vacpet	47.3 ± 0.3	31.1 ± 0.7	45.7 ± 0.5	39.6 ± 0.2	31.8 ± 0.2	45.4 ± 0.6
12	0vacpet	47.3 ± 0.3	30.6 ± 1.1	45.7 ± 0.5	39.6 ± 0.2	31.8 ± 0.2	45.4 ± 0.6

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

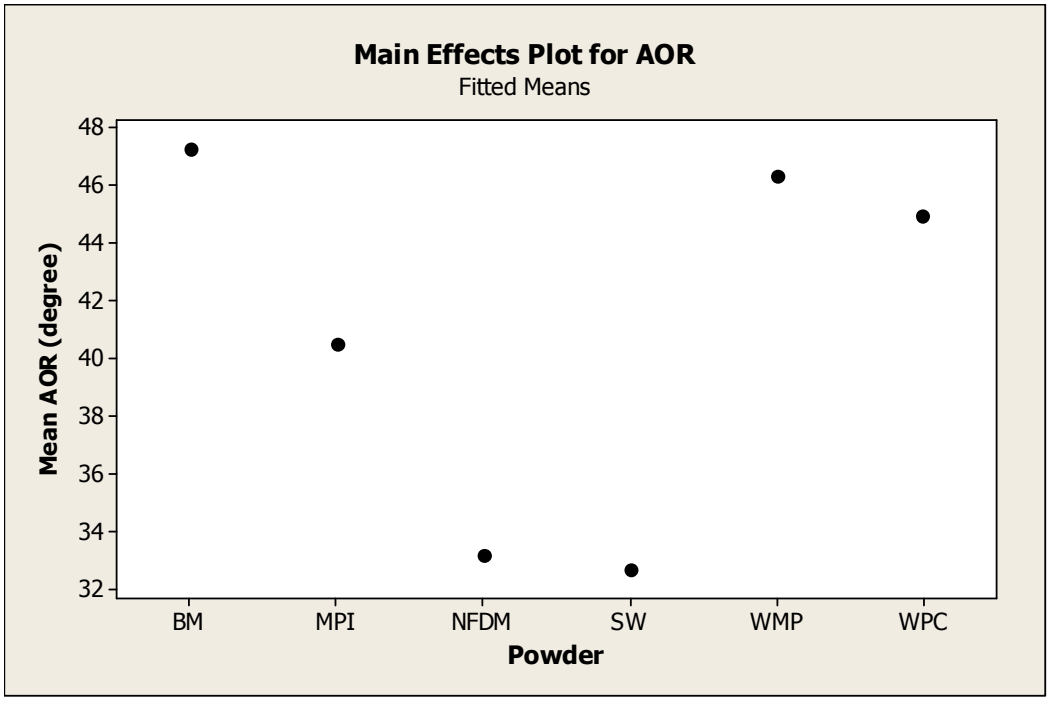

Figure 44: Main effects plots for Angle of repose (degree) versus powder type

Figure 44 shows the significant effect of powder type on the angle of repose. The differences observed should be mainly related to the differences in the physical and compositional properties of the powders such as particle size. It is known that particle size has a major influence on powder flowability; as particle size decreases, the surface area per unit mass of powder increases and this leads to reduced flowability. More contact surface area is available for cohesive forces, in particular, and frictional forces to resist flow. This can partially explain the high flowability (low AOR) observed in dairy powders with larger particle size such as SWP.

Flowability is important in handling the powder and discharging it from bins, silos, etc.

Particle shape, moisture content, and the surface compositions of powder particles are other affecting factors on powder flowability. It is shown that:

- Particle shape influences the surface contacts between particles (Fitzpatrick, 2005).
- An increase in moisture content results in an increase in liquid bridges and capillary forces acting between the powder particles, which eventually leads to reduced flowabiltiy. In addition, increased moisture content can soften (plasticize) the powder material, especially the water-soluble constituents, which would result in deformation of the powder giving a higher contact surface area (Scoville and Peleg, 1981). However, as no significant changes were observed in the moisture contents of the dairy powders studied in this project, the effect of moisture content on flowability can be ruled out. The changes of the powder moisture contents in this project are discussed later in this chapter.
- Fat on the surface of powders has a tendency to cause the particles to adhere to one another or agglomerate, deteriorating the flowability of the powders. Skim milk powder may flow more easily because the surface is made of lactose and protein with only a small amount of fat (18% surface fat), whereas dairy powders such as WMP and WPC flow poorly because the surfaces are largely made of fat (98%, and 53% surface fat, respectively) (Kim et al., 2005). This may explain some of the significant differences found in the flowability of different powders studied in this project; the higher flowability (smaller AOR) in low fat powders such as SWP and NFDM, comparing to the lower flowability (larger AOR) in high fat content powders such as WMP and WPC80.

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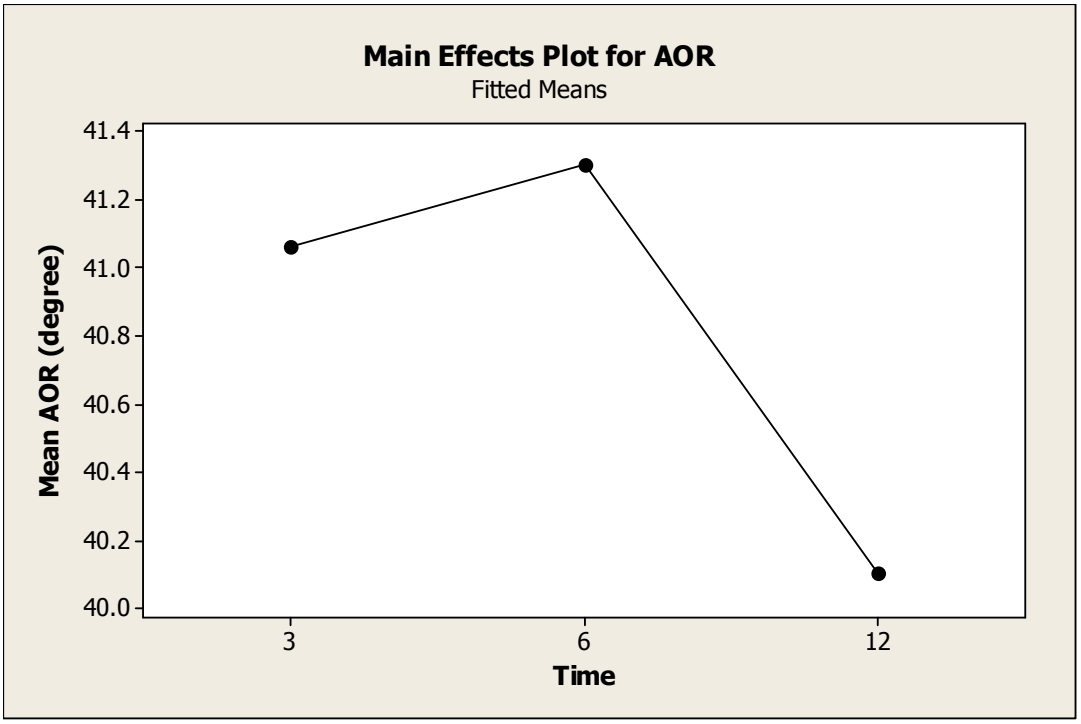

Figure 45: Main effects plots for Angle of repose (degree) versus storage time (month)

Figure 45 shows the significant effect of storage time on angle of repose with a downward trend over the storage time, which indicates an increase in flowability. This can be explained by the increased interparticle forces and increased particle size in the powders, caused by time consolidation of the powder over the storage time, as explicitly described earlier in this chapter.

5.7. Effect of vacuum packaging and storage time on compressibility

The data collected according to the method outlined in section 4.5.6., pertaining to compressibility, is shown in Appendix G. The statistical analysis of the mean compressibility was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there was not any statistically significant difference in mean compressibility of the powders due to the effect of vacuum pressure ($p = 0.970$). The interaction between storage time and vacuum pressure was not statistically significant ($p = 0.100$). However, there were statistically significant differences in compressibility, due to the effect of storage time ($p = 0.004 < 0.01$) and powder type ($p = 0.000 < 0.01$). The mean for each powder at different levels of treatment (vacuum pressure and storage time) are displayed in Table 10.

Table 10: Mean compressibility (mm) of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	14.194	12.46	16.365	19.439	6.46	24.634
3	99vacpet	14.574 ± 0.09	12.559 ± 0.05	15.383 ± 0.36	17.981 ± 0.25	5.880 ± 0.09	24.174 ± 0.31
3	50vacpet	14.908 ± 0.02	13.126 ± 0.61	15.625 ± 0.06	18.256 ± 0.54	6.643 ± 0.34	24.220 ± 0.11
3	25vacpet	13.986 ± 0.18	12.752 ± 0.19	15.064 ± 0.19	18.570 ± 0.41	6.198 ± 0.59	24.029 ± 1.32
3	0vacpet	14.293 ± 0.32	12.733 ± 0.58	14.362 ± 0.39	18.762 ± 0.30	6.803 ± 0.26	24.904 ± 0.92
6	99vacpet	14.094 ± 0.79	12.497 ± 0.20	14.922 ± 0.45	18.523 ± 0.28	5.696 ± 0.19	24.118 ± 0.30
6	50vacpet	14.224 ± 0.26	12.626 ± 0.01	15.015 ± 0.33	18.229 ± 0.22	6.549 ± 0.04	23.747 ± 0.54
6	25vacpet	14.040 ± 0.10	13.548 ± 0.74	14.185 ± 0.09	18.145 ± 0.27	6.518 ± 0.27	23.468 ± 0.38
6	0vacpet	14.336 ± 0.35	12.515 ± 0.26	14.631 ± 0.26	18.271 ± 0.01	6.312 ± 0.42	22.944 ± 0.17
12	99vacpet	14.404 ± 0.63	13.092 ± 0.30	15.112 ± 0.92	18.360 ± 0.32	6.434 ± 0.01	23.250 ± 0.75
12	50vacpet	13.975 ± 0.07	12.525 ± 0.41	14.056 ± 0.63	18.601 ± 0.48	6.011 ± 0.06	22.725 ± 0.27
12	25vacpet	14.508 ± 0.68	12.699 ± 0.13	14.842 ± 0.15	18.557 ± 0.02	6.631 ± 0.19	22.875 ± 1.18
12	0vacpet	14.264 ± 1.03	13.304 ± 0.53	14.580 ± 0.20	18.270 ± 0.12	6.115 ± 0.80	22.741 ± 0.70

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

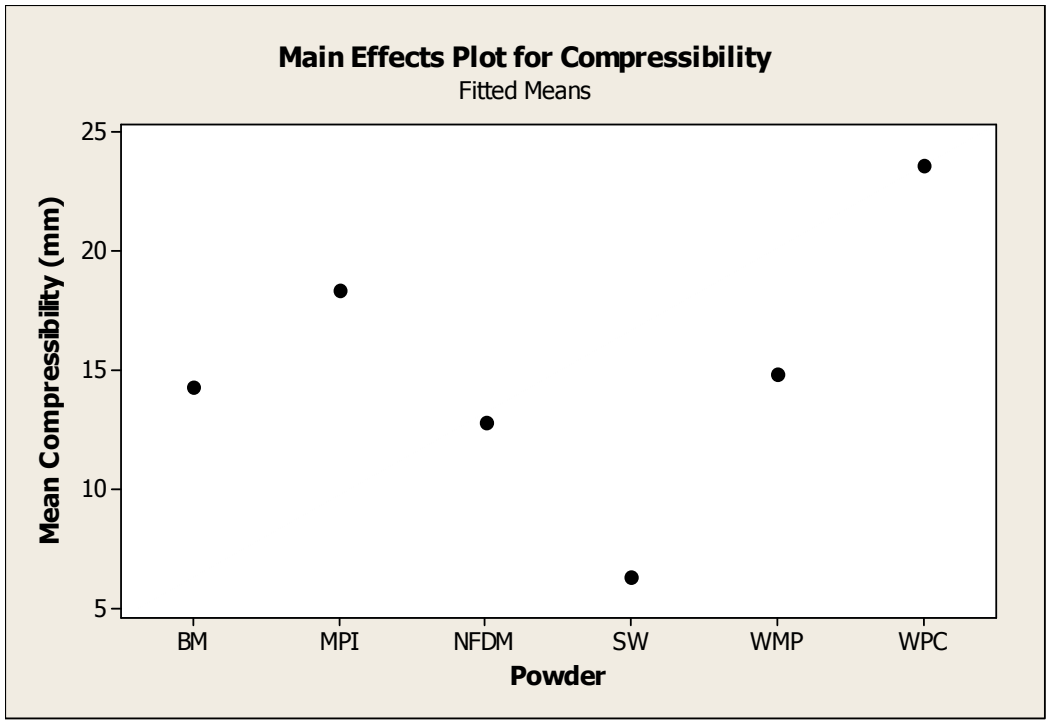

Figure 46: Main effects plots for compressibility versus powder type

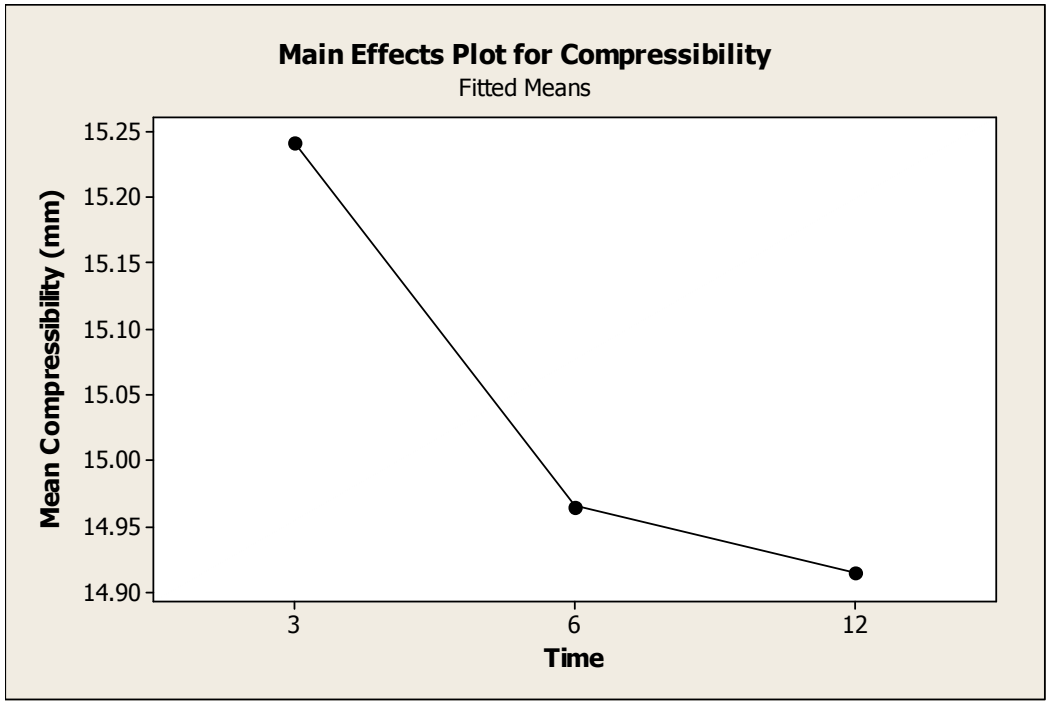

Figure 47: Main effects plots for compressibility versus storage time (month)

Figure 46 shows the significant effect of powder type on compressibility. The differences observed should be mainly related to the differences in the physical and compositional properties of the powders including: bulk density and particle size of powder particles.

- Bulk density also has a reverse correlation with compressibility. In fact, compressibility represents the change in bulk density of a powder under a known pressure (Yan et al., 2001). As low bulk density dairy powders, such as WPC80, have more voids among their particles, they are less resistant against the compression force, show higher compressibility, and get more compact under the compression force.
- Particle size has a reverse correlation with compressibility. In fact, finer particles result in higher compressibility, while larger particles cause lower compressibility (Yan and Barbosa-Canovas, 2000). This can explain the higher compressibility observed in a finer particle powder such as NFDM as well as the lower compressibility observed in a larger particle powder such as SWP.

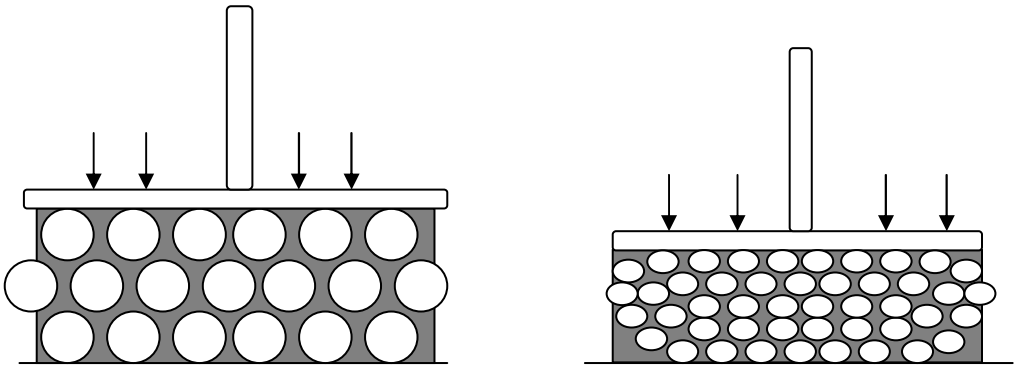

Figure 48: Schematic of the reverse correlation of particle size and compressibility. Under the same compression force, the powder with large particles (on the left) gets less compressed compared to the powder with small particles (on the right)

Figure 47 shows the significant effect of storage time on compressibility with a downward trend over the storage time. It can be explained by consolidation of powder over the storage time, which as explained earlier in this chapter leads to an increase in bulk density and particle size, and a decrease in compressibility, consequentially.

Compressibility is important in calculating the loading capacity of bags, silos, etc.

5.8. Effect of vacuum packaging and storage time on L-value

The data collected according to the method outlined in section 4.5.7., pertaining to the L- color value, is shown in Appendix H. The statistical analysis of the mean L- value was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there were statistically significant differences in mean L-value of the powders, due to the effects of powder type with ($p = 0.000$), and vacuum pressure with ($p = 0.003$). However, the effects of time and the interaction between storage time and vacuum pressure were not statistically significant with ($p = 0.020$) and ($p = 0.994$), respectively. The means for all the powders at different levels of treatment (vacuum pressure and storage time) are displayed in Table 11.

Table 11: Mean L-value of the powders tested at different storage times (0, 3, 6, 12 months)

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Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	90.21	90.88	90.76	87.86	87.56	87.42
3	99vacpet	90.18 ± 0.30	90.84 ± 0.18	90.59 ± 0.01	87.79 ± 0.68	87.13 ± 0.16	87.53 ± 0.40
3	50vacpet	90.23 ± 0.13	90.69 ± 0.19	90.27 ± 0.48	88.09 ± 0.94	87.21 ± 0.16	87.33 ± 0.30
3	25vacpet	90.16 ± 0.11	90.71 ± 0.23	90.42 ± 0.14	88.01 ± 0.23	87.28 ± 0.28	87.65 ± 0.37
3	0vacpet	90.08 ± 0.18	90.86 ± 0.96	89.87 ± 0.45	87.77 ± 0.52	86.94 ± 0.36	87.32 ± 0.58
6	99vacpet	89.92 ± 1.35	90.97 ± 0.51	90.14 ± 0.86	87.69 ± 0.37	87.25 ± 0.18	87.47 ± 0.42
6	50vacpet	89.83 ± 0.35	90.83 ± 0.26	90.28 ± 0.27	87.62 ± 0.44	87.11 ± 0.34	87.35 ± 0.10
6	25vacpet	89.96 ± 0.44	91.07 ± 0.04	90.16 ± 0.13	87.78 ± 0.57	87.15 ± 0.12	87.29 ± 0.31
6	0vacpet	90.13 ± 0.16	90.72 ± 0.11	89.34 ± 0.27	87.46 ± 0.53	87.05 ± 0.12	86.88 ± 0.34
12	99vacpet	89.97 ± 0.21	90.73 ± 0.11	90.32 ± 0.35	87.81 ± 0.01	87.13 ± 0.35	87.17 ± 0.25
12	50vacpet	90.20 ± 0.05	90.63 ± 0.92	90.16 ± 0.30	87.53 ± 0.00	87.06 ± 0.18	87.05 ± 0.29
12	25vacpet	90.02 ± 0.32	90.72 ± 0.42	90.27 ± 0.17	87.54 ± 0.37	87.16 ± 0.32	87.38 ± 0.08
12	0vacpet	89.92 ± 0.32	90.49 ± 0.52	88.86 ± 0.56	87.63 ± 0.44	87.25 ± 0.30	87.15 ± 0.32

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

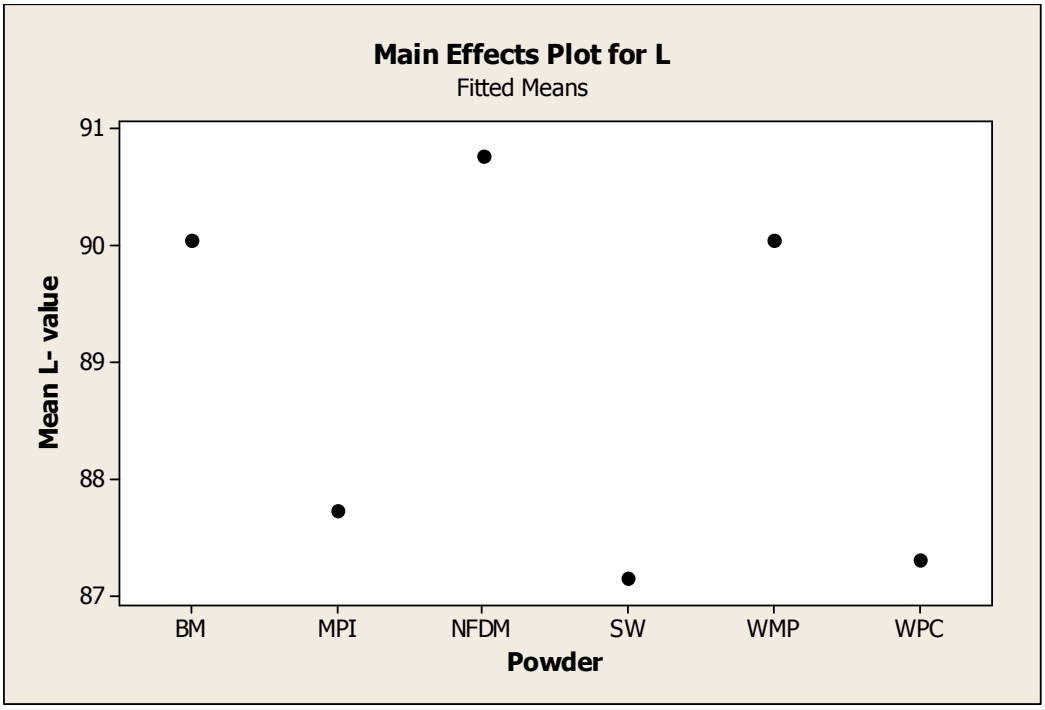

Figure 49: Main effects plot for L-value versus powder type

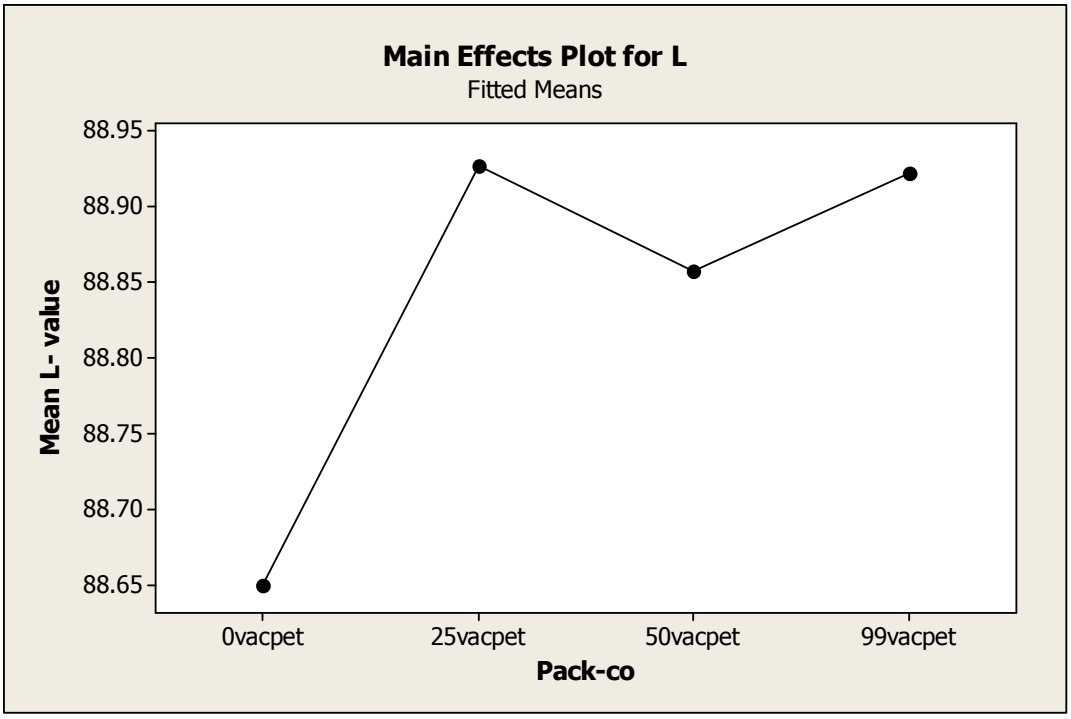

Figure 50: Main effects plot for L-value versus packaging conditions (Pack-co)

Figure 49 shows the significant effect of powder type on L-value. The differences observed should be mainly related to the differences in the compositional properties and processing specifications of the powders.

Figure 50 shows the significant effect of vacuum pressure on L-value with an upward trend as the vacuum pressure increases. L- color value, is an indicator of powder's whiteness/ darkness, hence observing a lower mean L-value for control (air packaged) powders indicates that vacuum packaging prevents darkening of the powders. This result is in agreement with the other research that shows nitrogen-flushed whole milk powder (WMP) samples are whiter and have lower hexanal (a marker of lipid oxidation) concentratins compared to the atmospheric packaged WMP samples (Lloyd et al, 2009). It is expected that vacuum packaging has a similar effect on preventing the lipid oxidation, hence the hexanal concentration in dairy powders; however, the validity of such an assumption yet needs to be investigated.

5.9. Effect of vacuum packaging and storage time on a-value

The data collected according to the method outlined in section 4.5.7, pertaining to the a- color value, is shown in Appendix I. The statistical analysis of the mean a- value was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there were statistically significant differences in mean a-value of the powders, due to the effects of powder type with ($p = 0.000$), and vacuum pressure ($p = 0.005$). However, the effects of time and the interaction between storage time and vacuum pressure were not statistically significant with ($p = 0.256$) and ($p = 0.745$),

respectively. The means for all the powders at different levels of treatment (vacuum pressure and storage time) are displayed in Table 12.

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Table 12: Mean a-value of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	-3.49	-2.52	-1.95	-0.43	-1.78	0.38
3	99vacpet	-3.51 ± 0.07	-2.39 ± 0.04	-1.97 ± 0.01	-0.43 ± 0.02	-1.85 ± 0.02	0.37 ± 0.01
3	50vacpet	-3.53 ± 0.02	-2.41 ± 0.08	-1.94 ± 0.02	-0.43 ± 0.01	-1.85 ± 0.02	0.37 ± 0.01
3	25vacpet	-3.48 ± 0.04	-2.42 ± 0.06	-1.98 ± 0.01	-0.42 ± 0.04	-1.86 ± 0.02	0.36 ± 0.01
3	0vacpet	-3.35 ± 0.08	-2.37 ± 0.03	-1.91 ± 0.03	-0.44 ± 0.00	-1.85 ± 0.02	0.37 ± 0.01
6	99vacpet	-3.51 ± 0.05	-2.43 ± 0.04	-1.97 ± 0.01	-0.45 ± 0.03	-1.84 ± 0.00	0.37 ± 0.01
6	50vacpet	-3.52 ± 0.02	-2.38 ± 0.00	-1.97 ± 0.01	-0.43 ± 0.03	-1.84 ± 0.01	0.37 ± 0.01
6	25vacpet	-3.49 ± 0.03	-2.42 ± 0.05	-1.95 ± 0.04	-0.44 ± 0.01	-1.86 ± 0.02	0.35 ± 0.03
6	0vacpet	-3.47 ± 0.03	-2.40 ± 0.03	-1.89 ± 0.03	-0.43 ± 0.03	-1.86 ± 0.01	0.35 ± 0.02
12	99vacpet	-3.54 ± 0.04	-2.39 ± 0.06	-1.98 ± 0.01	-0.42 ± 0.04	-1.85 ± 0.02	0.36 ± 0.01
12	50vacpet	-3.55 ± 0.01	-2.43 ± 0.08	-1.96 ± 0.01	-0.45 ± 0.01	-1.85 ± 0.01	0.35 ± 0.03
12	25vacpet	-3.54 ± 0.04	-2.41 ± 0.06	-1.94 ± 0.04	-0.44 ± 0.02	-1.84 ± 0.02	0.36 ± 0.01
12	0vacpet	-3.48 ± 0.04	-2.44 ± 0.06	-1.87 ± 0.01	-0.45 ± 0.01	-1.83 ± 0.01	0.37 ± 0.01

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

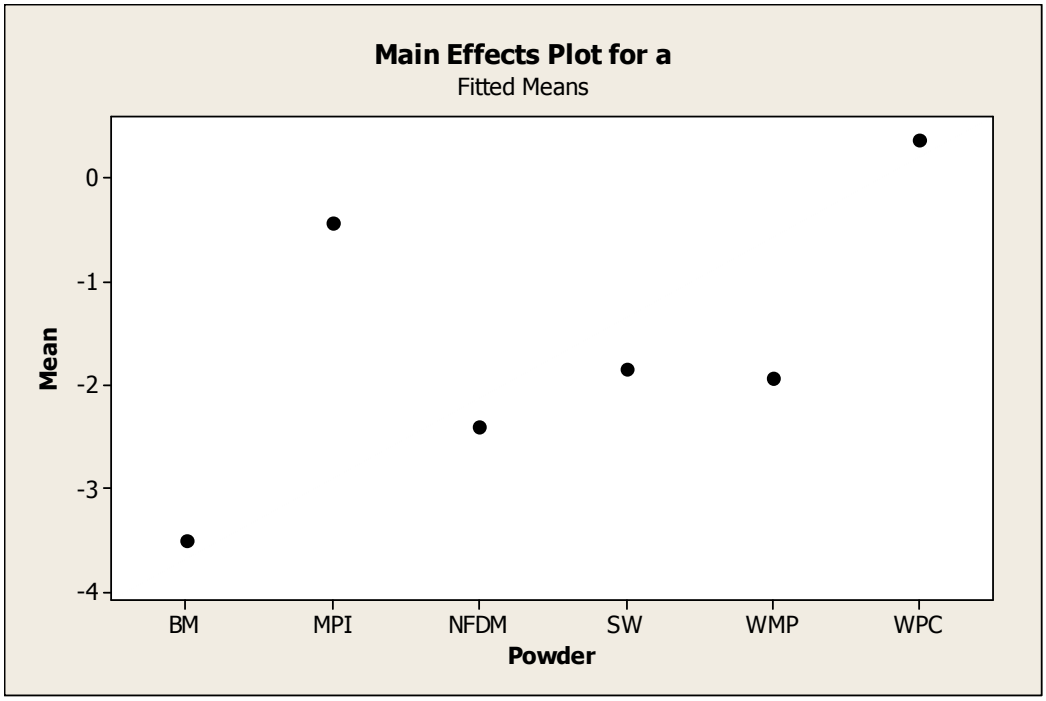


Figure 51: Main effects plot for a-value versus powder type

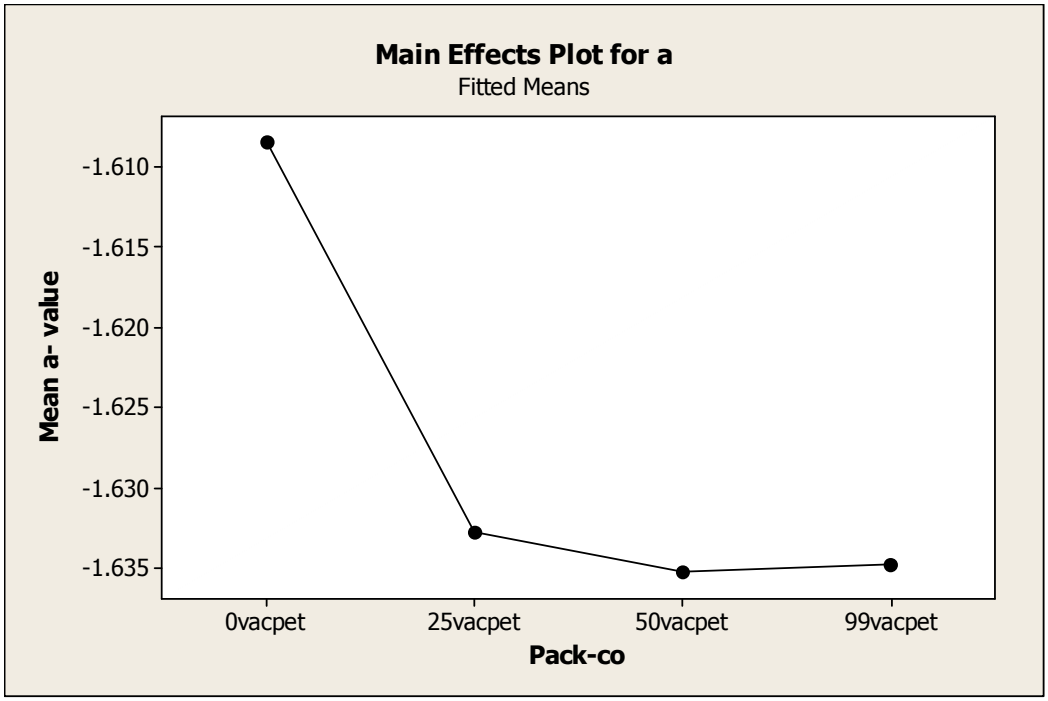


Figure 52: Main effects plot for a-value versus packaging conditions (Pack-co)

Figure 51 shows the significant effect of powder type on a-value. The differences observed should be mainly related to the differences in the compositional properties and processing specifications of the powders.

Figure 52 shows the significant effect of vacuum pressure on a-value with a downward trend as the vacuum pressure increases. a- color value, is an indicator of powder's redness/ greenness, hence observing a higher mean a-value for control (air packaged) powders indicates that vacuum packaging prevents the reactions that increase redness in the powders. As this effect seems to be interestingly more dramatic in WMP, which is a high fat containing powder, it might have been caused by oxidation of lipids, which is more likely to happen at higher oxygen levels in the control (air packaged) powders.

5.10. Effect of vacuum packaging and storage time on b-value

The data collected according to the method outlined in section 4.5.7., pertaining to the b- color value, is shown in Appendix J. The statistical analysis of the mean b- value on HunterLab Ultra-scan XE spectrophotometer was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there were statistically significant differences in mean b-value of the powders, due to the effects of powder type with ($p = 0.006$), and vacuum pressure with ($p = 0.001$). However, the effects of time and the interaction between storage time and vacuum pressure were not statistically significant with ($p = 0.689$) and ($p = 0.749$), respectively. The means for all the powders at different levels of treatment (vacuum pressure and storage time) are displayed in Table 13.

Table 13: Mean b-value of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	17.68	11.53	16.69	12.84	21.48	13.88
3	99vacpet	17.51 ± 0.10	11.58 ± 0.14	16.61 ± 0.09	12.76 ± 0.17	21.16 ± 0.60	13.78 ± 0.20
3	50vacpet	17.56 ± 0.25	11.72 ± 0.01	16.68 ± 0.11	12.65 ± 0.16	21.27 ± 0.85	13.66 ± 0.40
3	25vacpet	17.67 ± 0.05	11.56 ± 0.04	16.73 ± 0.13	12.73 ± 0.13	21.43 ± 0.42	13.69 ± 0.10
3	0vacpet	17.86 ± 0.14	11.56 ± 0.22	17.17 ± 0.08	12.35 ± 0.08	21.39 ± 0.34	13.78 ± 0.10
6	99vacpet	17.69 ± 0.22	11.51 ± 0.62	16.46 ± 0.03	12.88 ± 0.02	21.09 ± 0.28	13.78 ± 0.16
6	50vacpet	17.48 ± 0.13	11.47 ± 0.03	16.56 ± 0.22	12.78 ± 0.18	21.44 ± 0.11	13.85 ± 0.33
6	25vacpet	17.58 ± 0.17	11.64 ± 0.12	16.67 ± 0.03	12.73 ± 0.12	21.32 ± 0.23	13.72 ± 0.02
6	0vacpet	17.93 ± 0.06	11.54 ± 0.45	17.32 ± 0.16	12.84 ± 0.01	21.27 ± 0.21	14.23 ± 0.13
12	99vacpet	17.54 ± 0.18	11.43 ± 0.41	16.57 ± 0.16	12.77 ± 0.13	21.70 ± 0.81	13.73 ± 0.21
12	50vacpet	17.71 ± 0.06	11.59 ± 0.16	16.64 ± 0.17	12.64 ± 0.18	21.44 ± 0.17	13.45 ± 0.16
12	25vacpet	17.54 ± 0.07	11.63 ± 0.30	16.81 ± 0.25	12.56 ± 0.03	21.32 ± 0.12	13.68 ± 0.22
12	0vacpet	17.97 ± 0.08	11.51 ± 0.19	17.24 ± 0.13	12.73 ± 0.06	21.36 ± 0.16	14.31 ± 0.05

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

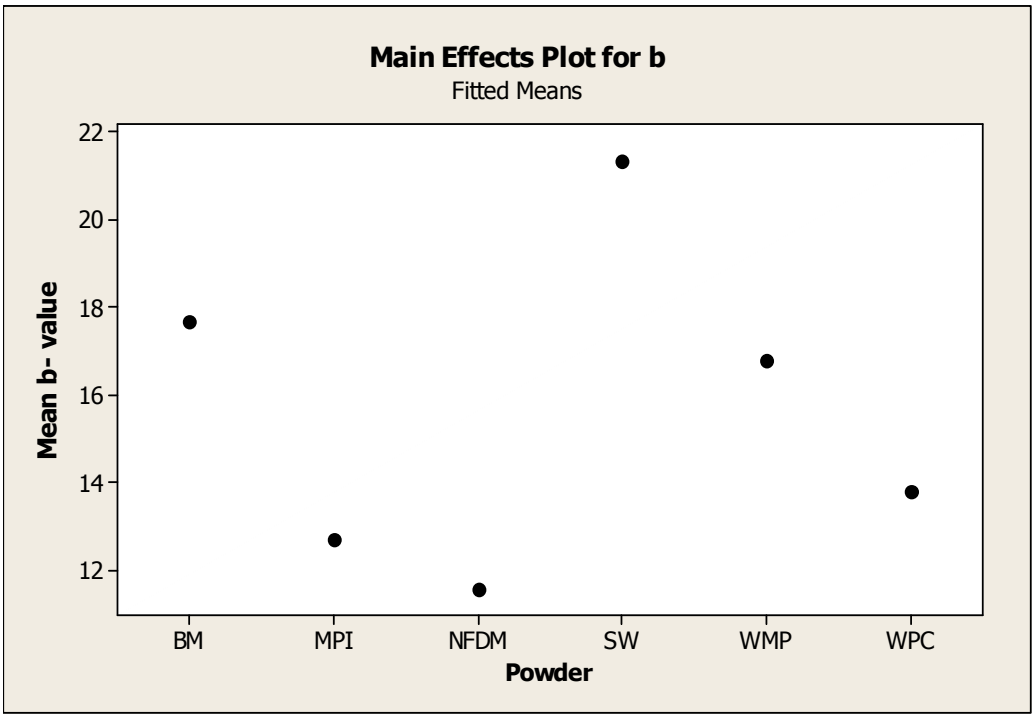


Figure 53: Main effects plot for b-value versus powder type

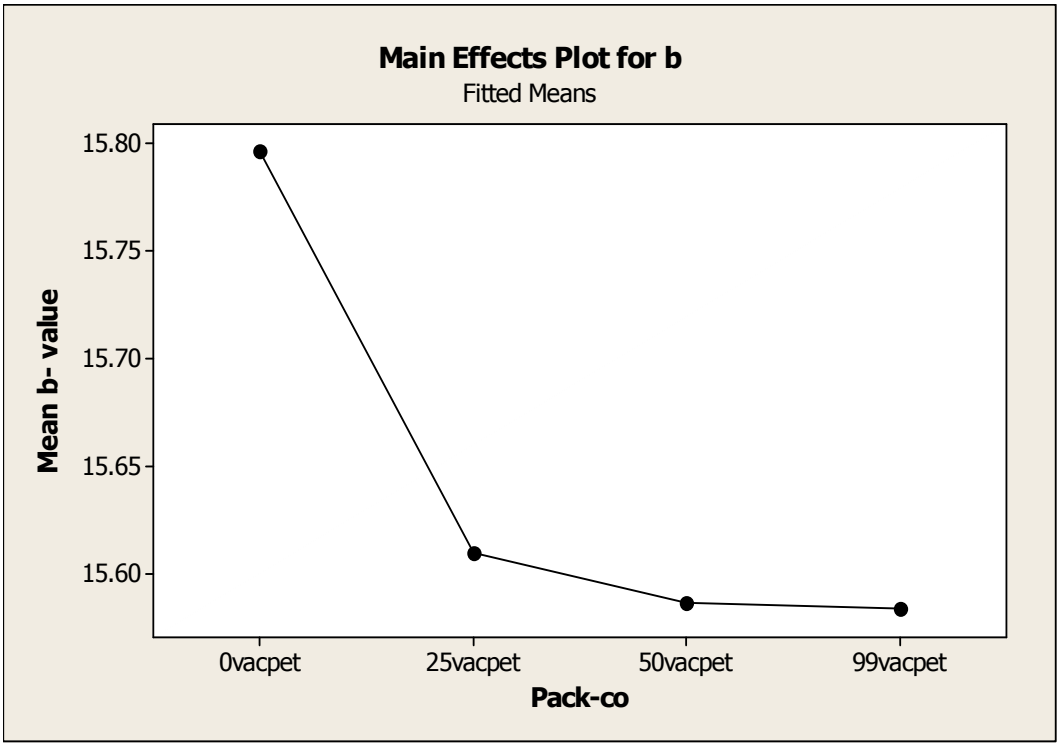


Figure 54: Main effects plot for b-value versus packaging conditions (Pack-co)

Figure 53 shows the significant effect of powder type on b-value. The differences observed should be mainly related to the differences in the compositional properties and processing specifications of the powders.

Figure 54 shows the significant effect of vacuum pressure on a-value with a downward trend as the vacuum pressure increases. b- color value, is an indicator of powder's yellowness/ blueness, hence observing a higher mean b-value for control (air packaged) powders indicates that vacuum packaging prevents the reactions that increase yellowness in the powders. As this effect is interestingly more dramatic in BM, WPC, and especially WMP, all high fat containing powders, it have been caused by oxidation of lipids, which is more likely to happen at higher oxygen levels in the control (air packaged) powders.

In conclusion, the color analyses of the powders showed that atmospheric packaged powders, turned darker, redder, and more yellow during storage. This effect is likely correlated with color changes due to lipid oxidation, as it was more dramatic in higher fat containing powders such as BMP, WPC, and especially WMP and it is in agreement with previous research showing the same color changes in milk powders, due to oxidation during storage (Nielsen et al., 1997b; Nielsen et al., 1997a; Stapelfeldt et al., 1997).

Color is an important aspect of dairy powder's appearance and influences the powder functionality as well as consumers' perception of powder quality.

5.11. Effect of vacuum packaging and storage time on moisture content

The data collected according to the method outlined in section 4.3.2., pertaining to the moisture content, is shown in Appendix K $_{(1\ \&\ 2)}$. The statistical analysis of the moisture content was conducted using the GLM command in Minitab according to section 4.2.2.

At $\alpha = 0.01$, there was not any statistically significant differences in mean moisture content of the powders, due to the effects of: time ($p = 0.847$), powder type ($p = 0.052$), and vacuum pressure ($p = 0.912$). The interaction between storage time and vacuum pressure was not statistically significant ($p = 0.878$). The means for all the powders at different levels of treatment (vacuum pressure and storage time) are displayed in Table 14..

It is important to notice that despite the differences observed in the initial moisture content of the powders, the p-value for powder type was not significant ($p = 0.052$). To show the differences due to powder type, the statistical test was reconducted after removing the data for "time 0". It resulted in a significant p-value = 0.000 for powder type the insignificant p-values of 0.847, 0.912, and 0.878 for the effects of storage time, vacuum pressure, and the interaction between storage time and vacuum pressure, respectively.

Table 14: Mean moisture content (%) of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	3.46	3.39	4.29	5.42	3.68	5.47
3	99vacpet	3.39 ± 0.06	3.28 ± 0.04	4.11 ± 0.05	5.26 ± 0.09	3.54 ± 0.28	5.33 ± 0.13
3	50vacpet	3.53 ± 0.03	3.31 ± 0.04	4.12 ± 0.06	5.21 ± 0.09	3.57 ± 0.25	5.36 ± 0.16
3	25vacpet	3.23 ± 0.06	3.22 ± 0.05	4.04 ± 0.04	5.41 ± 0.17	3.53 ± 0.21	5.35 ± 0.33
3	0vacpet	3.19 ± 0.02	3.21 ± 0.07	4.13 ± 0.04	5.37 ± 0.13	3.63 ± 0.37	5.52 ± 0.18
6	99vacpet	3.33 ± 0.18	3.18 ± 0.25	4.08 ± 0.06	5.45 ± 0.06	3.53 ± 0.15	5.38 ± 0.17
6	50vacpet	3.13 ± 0.06	3.33 ± 0.08	4.25 ± 0.18	5.33 ± 0.13	3.63 ± 0.35	5.32 ± 0.05
6	25vacpet	3.26 ± 0.03	3.24 ± 0.11	4.26 ± 0.24	5.46 ± 0.09	3.54 ± 0.20	5.35 ± 0.19
6	0vacpet	3.27 ± 0.06	3.35 ± 0.04	4.15 ± 0.13	5.38 ± 0.20	3.58 ± 0.17	5.34 ± 0.19
12	99vacpet	3.37 ± 0.22	3.14 ± 0.06	4.20 ± 0.01	5.31 ± 0.11	3.57 ± 0.25	5.37 ± 0.12
12	50vacpet	3.19 ± 0.08	3.28 ± 0.06	4.18 ± 0.04	5.42 ± 0.16	3.55 ± 0.11	5.32 ± 0.07
12	25vacpet	3.46 ± 0.04	3.32 ± 0.10	4.14 ± 0.10	5.39 ± 0.23	3.68 ± 0.16	5.31 ± 0.18
12	0vacpet	3.12 ± 0.04	3.23 ± 0.08	4.17 ± 0.11	5.54 ± 0.02	3.57 ± 0.05	5.39 ± 0.32

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

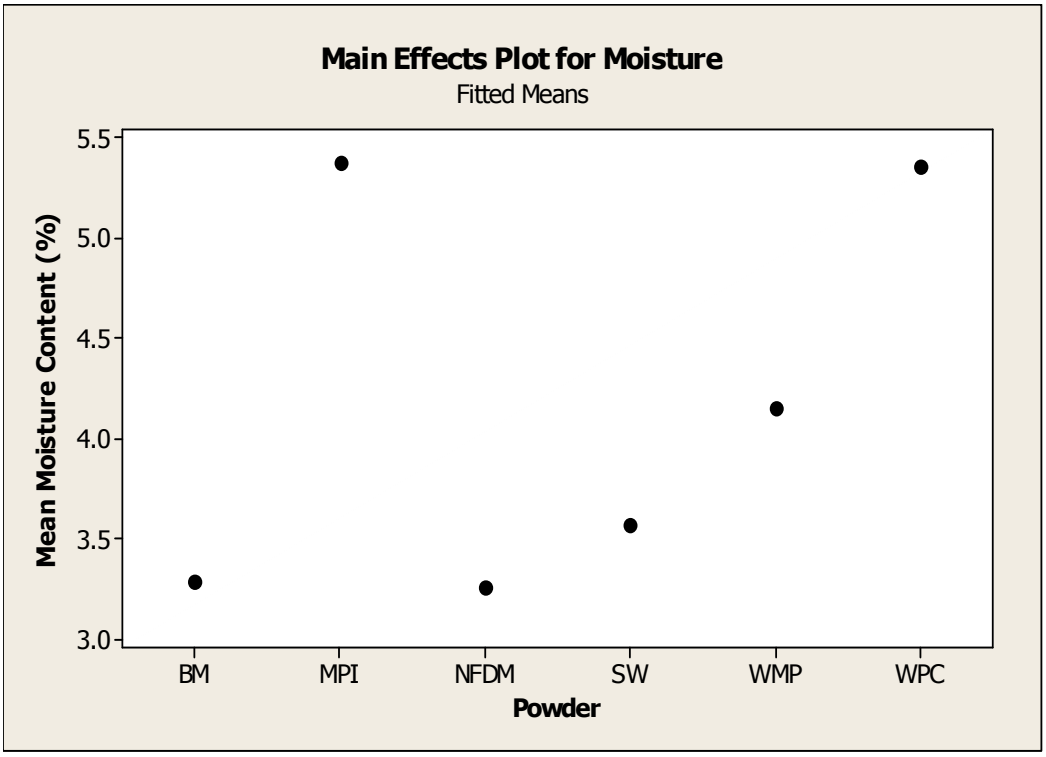


Figure 55: Main effects plot for moisture content versus powder type

No significant difference in moisture content of the powders is a good indicator of low MVPR (moisture vapor transmission rate) of the packaging material and good seal integrity of the bags during the storage time.

5.12. Effect of vacuum packaging and storage time on solubility

The data collected according to the method outlined in section 4.6.1., pertaining to solubility, is shown in Appendix L. The statistical analysis of solubility was conducted using the GLM command in Minitab according to section 4.2.2.

There was not any significant differences (neither positive, nor negative) in the mean moisture content of the powders, due to the effects of: time and vacuum pressure. The interaction between storage time and vacuum pressure was not statistically

significant. However there was a significant effect of powder type on the solubility of the powders tested. The means for all the powders at different levels of treatment (vacuum pressure and storage time) are displayed in Table 15.

It is important to notice that although the mean solubility of the 6 different powders tested were different from each other, it was impossible to calculate a p-value for this difference, as there was no standard deviation within each powder type.

Table 15: Mean solubility of the powders tested at different storage times (0, 3, 6, 12 months). Means and standard deviations were calculated from two measurements.

Time	Pack-co	BM	NFDM	WMP	MPI	SW	WPC
0	Kraft	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
3	99vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
3	50vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
3	25vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
3	0vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
6	99vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
6	50vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
6	25vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
6	0vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
12	99vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
12	50vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
12	25vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00
12	0vacpet	0.12 ± 0.00	0.10 ± 0.00	0.10 ± 0.00	1.50 ± 0.00	0.00 ± 0.00	0.25 ± 0.00

BM = buttermilk powder; NFDM = nonfat dry milk; WMP = whole milk powder;
MPI = milk protein isolate; SW = sweet whey powder; WPC = whey protein concentrate80;

Kraft = no vacuum, received from company in kraft paper bag
99vacpet = 99% vacuum, packaged in multilayer bag
50vacpet = 50% vacuum, packaged in multilayer bag
25vacpet = 25% vacuum, packaged in multilayer bag
0vacpet = no vacuum, packaged in multilayer bag
Multilayer bags consisted of: polyethylene terephthalate, aluminum and linear low density polyethylene

0 = right after receiving from company; 3 = after 3 months storage;
6 = after 6 months storage; 12 = after 12 months storage

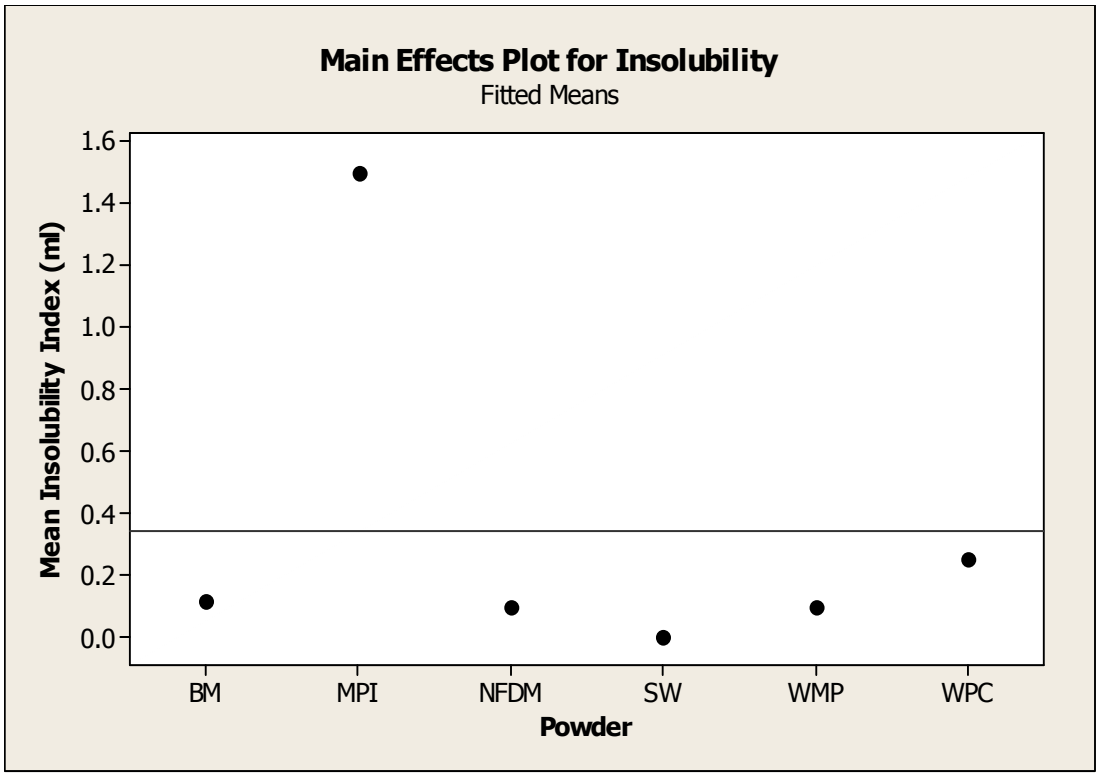


Figure 56: Main effects plot for insolubility index versus powder type

Solubility is a key functional property that influences other functional properties of dairy powders, including viscosity, foaming, emulsifying, etc.

5.13. Effect of vacuum packaging and storage time on surface morphology

Scanning electron micrographs of the powders were taken according to the method outlined in section 4.5.8. The micrographs below show the shape and surface morphology of the particles in different powders.

Surface morphology has an influence on the physical and functional properties of dairy powders.

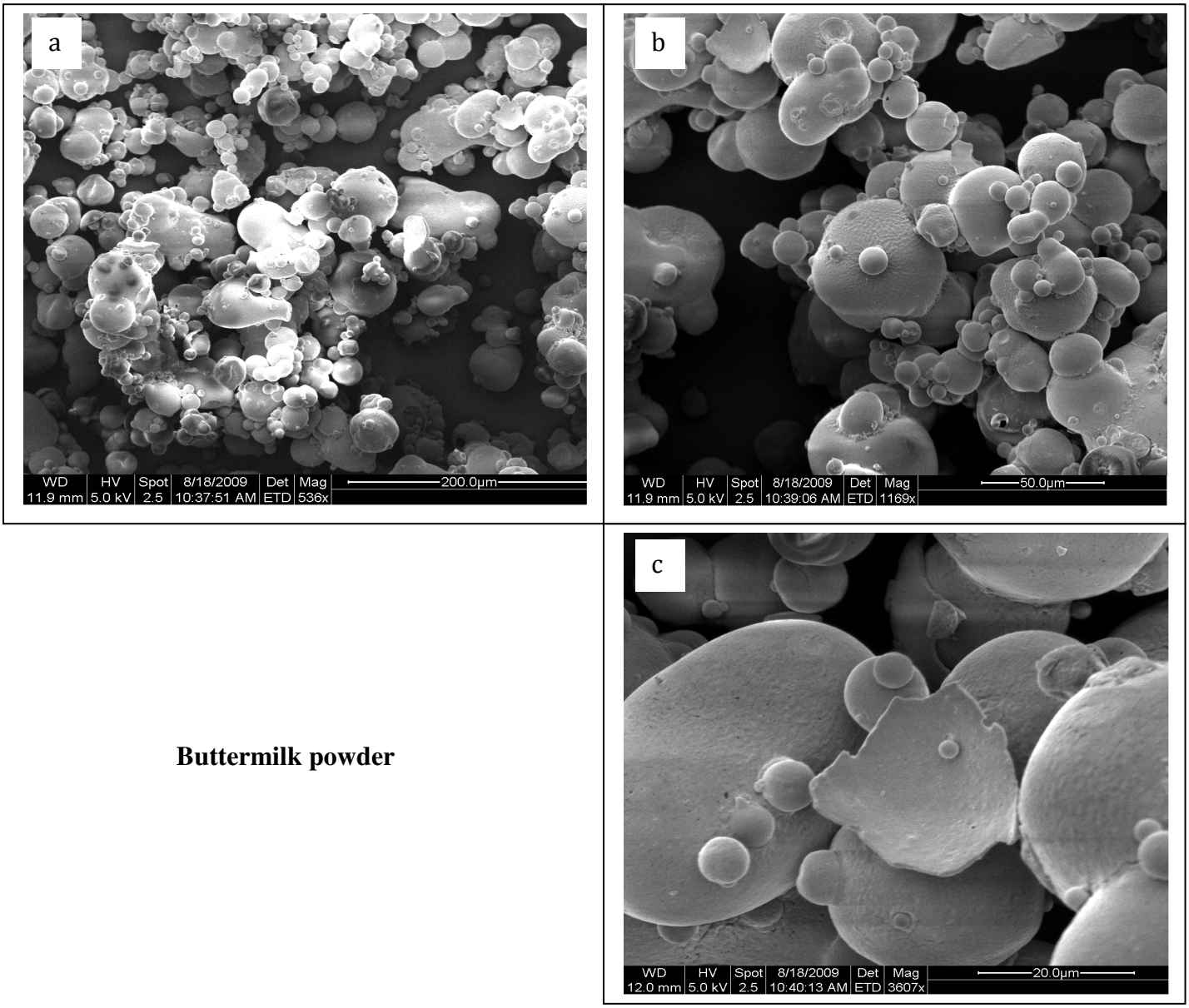
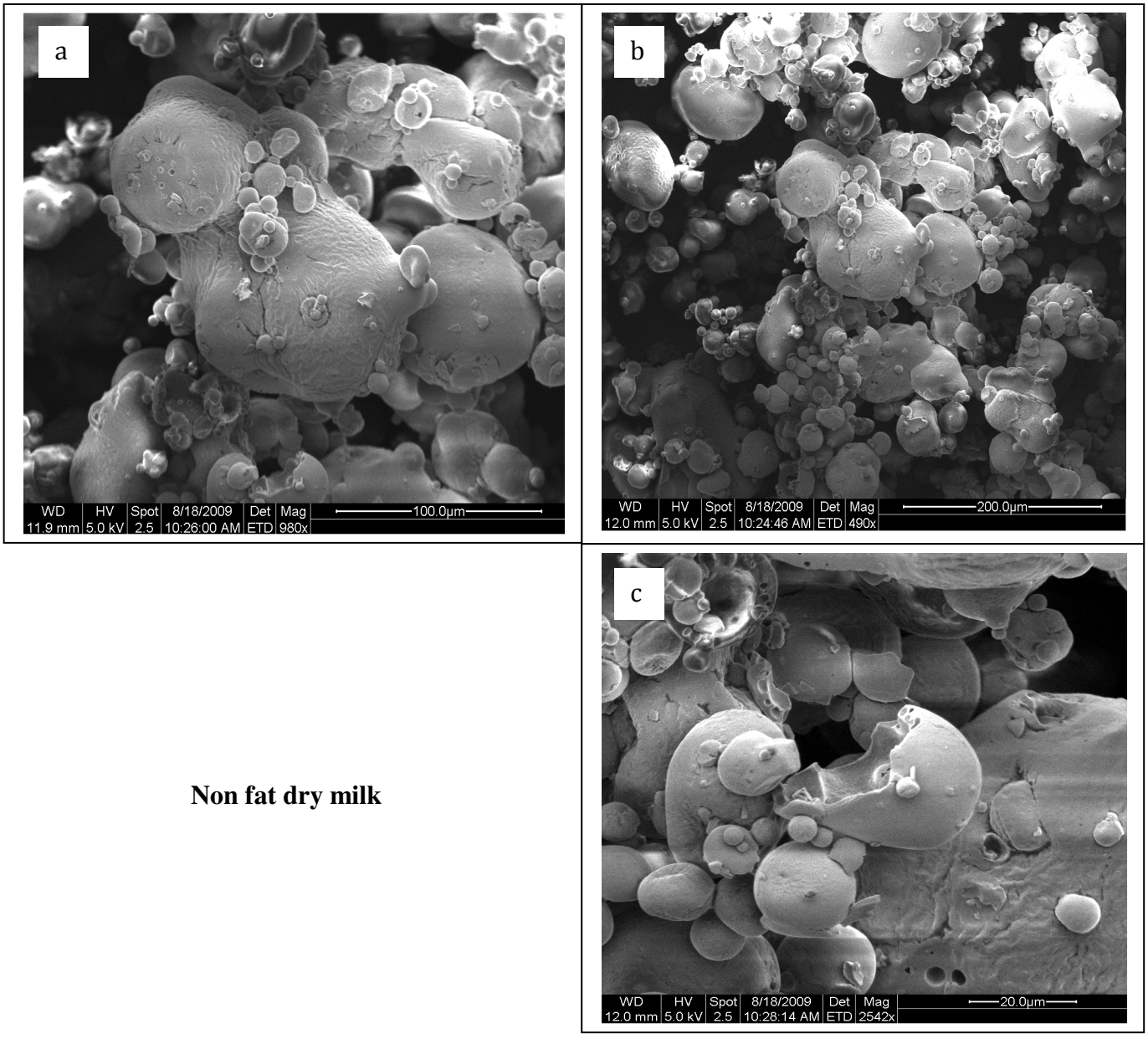
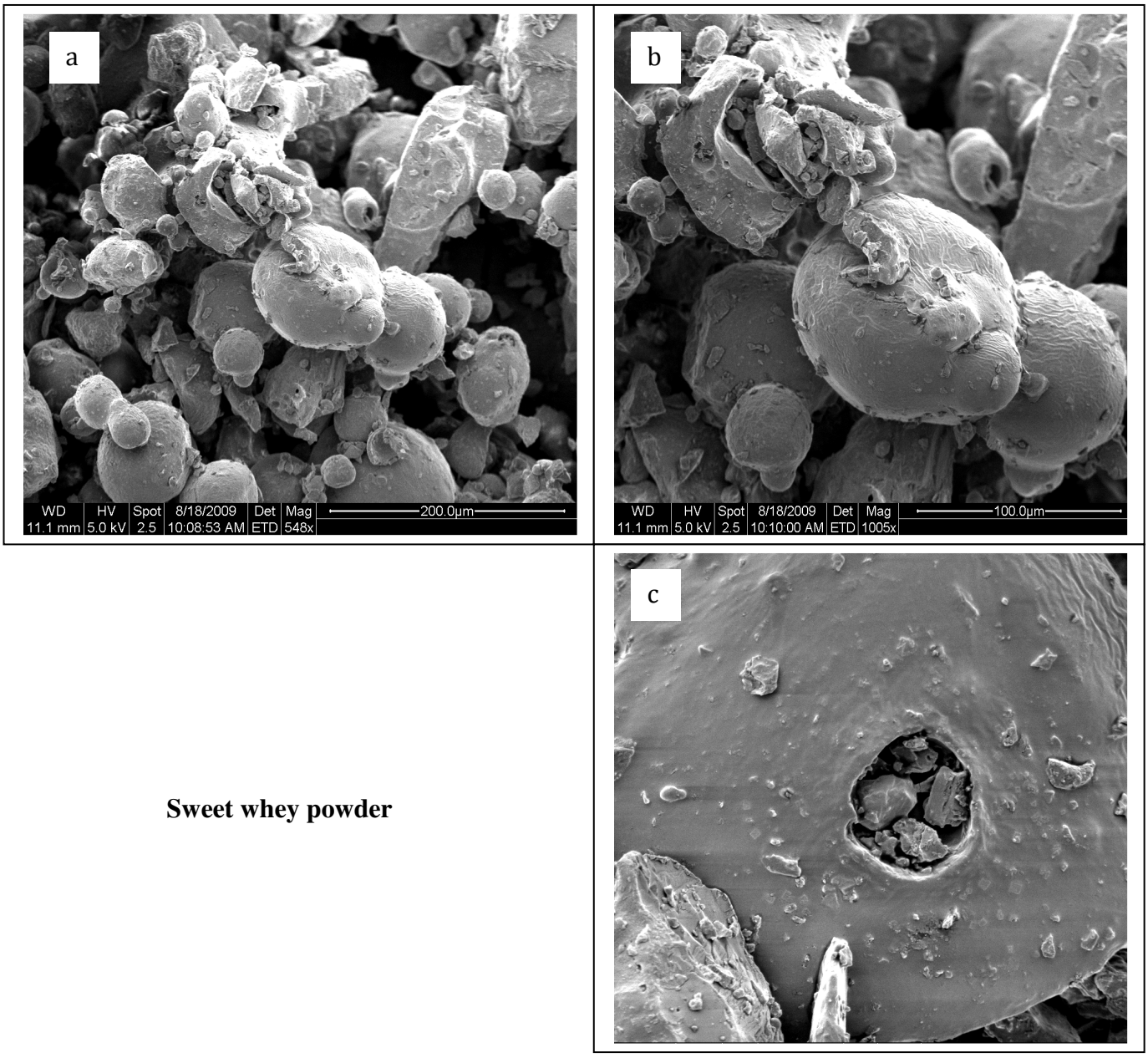


Figure 57: Scanning electron micrographs of buttermilk powder. The characteristics shown include: (a) and (b) shallow wrinkles (on the surface of large particles), (c) a few egg shell like structures.



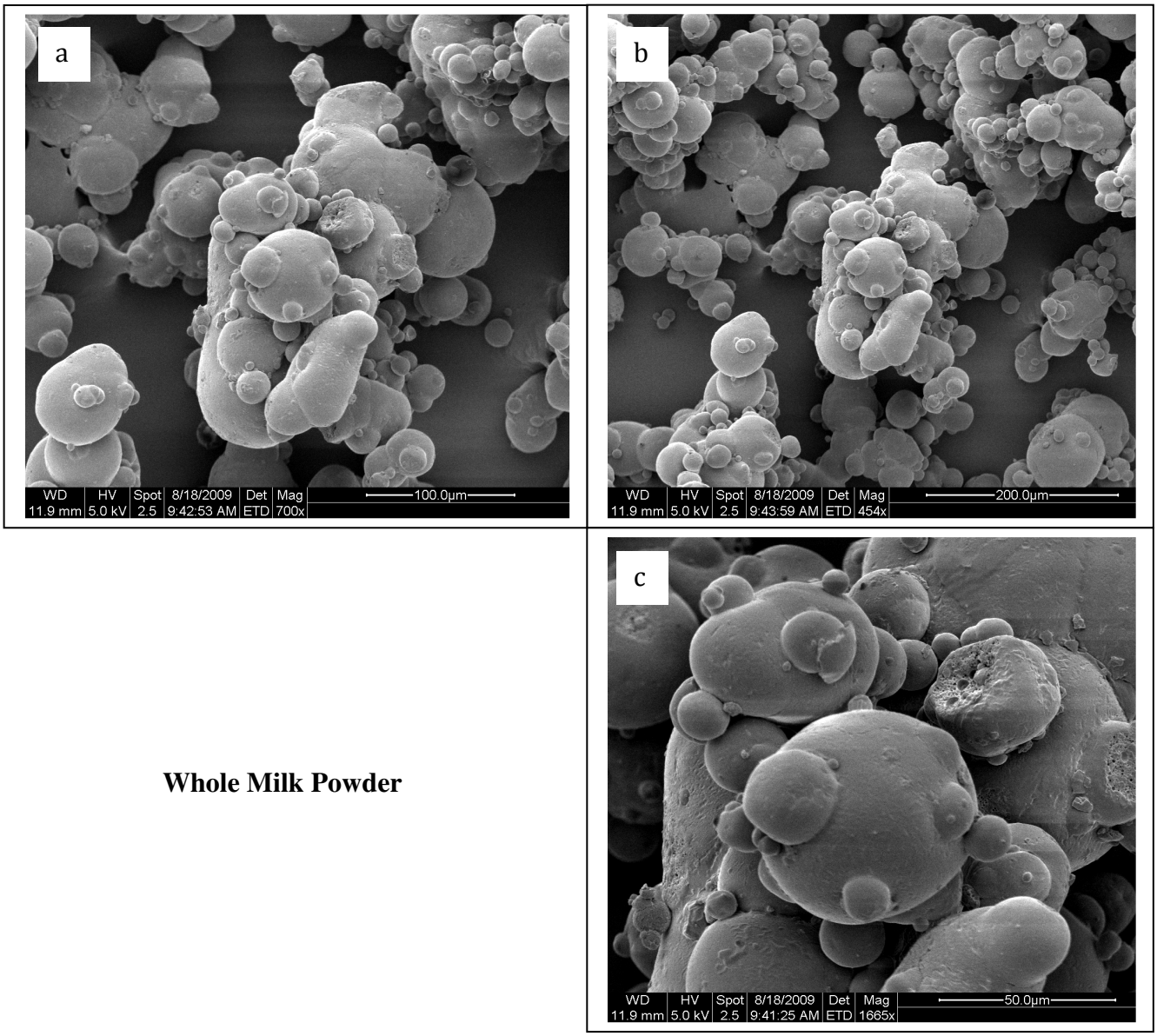
Non fat dry milk

Figure 58: Scanning electron micrographs of non fat dry milk powder. The characteristics shown include: (a) and (b) some shallow wrinkles and minute pores on the surface of particles. (c) a few broken particles.



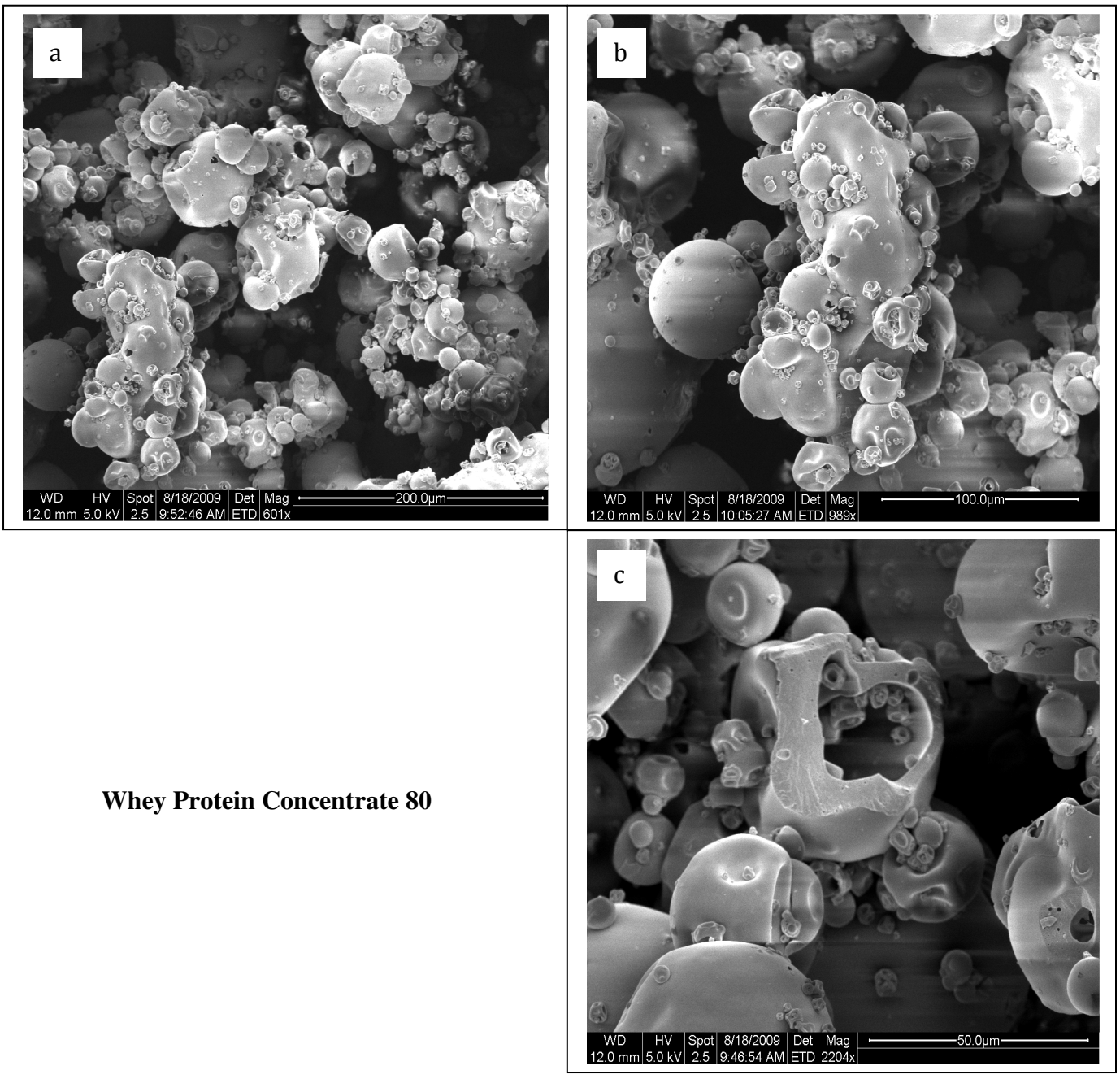
Sweet whey powder

Figure 59: Scanning electron micrographs of sweet whey powder. The characteristics shown include: (a), (b), and (c) many particles filled with holes and a honeycomb structure.



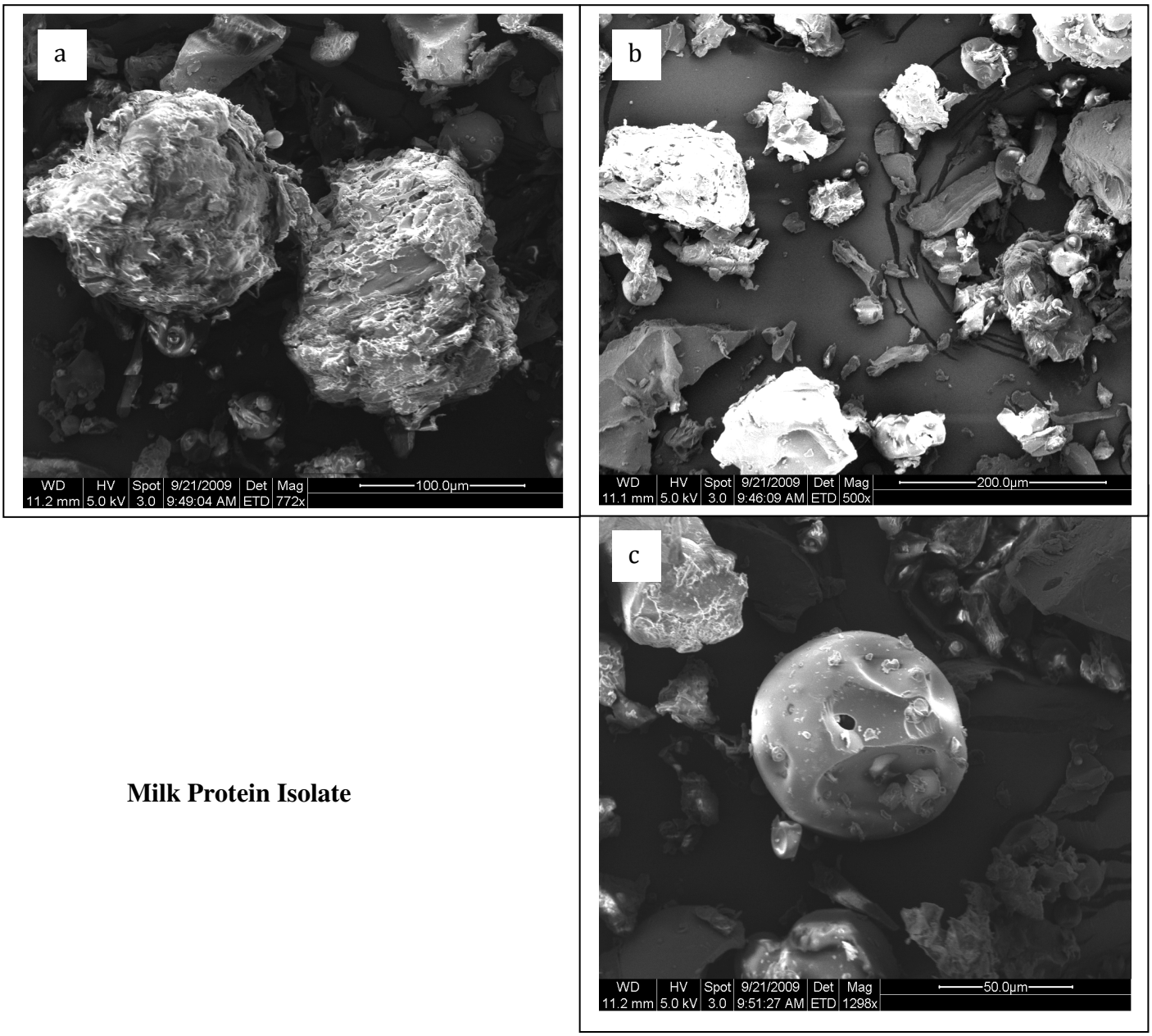
Whole Milk Powder

Figure 60: Scanning electron micrographs of whole milk powder. The characteristics shown include: (a), (b) and (c) relatively smooth particle surface and a few broken particles



Whey Protein Concentrate 80

Figure 61: Scanning electron micrographs of whey protein concentrate 80. The characteristics shown include: (a), (b), and (c) particles with a smooth surface and shallow or deep dimples with some small particles infusing in the deep dimples of large particles.



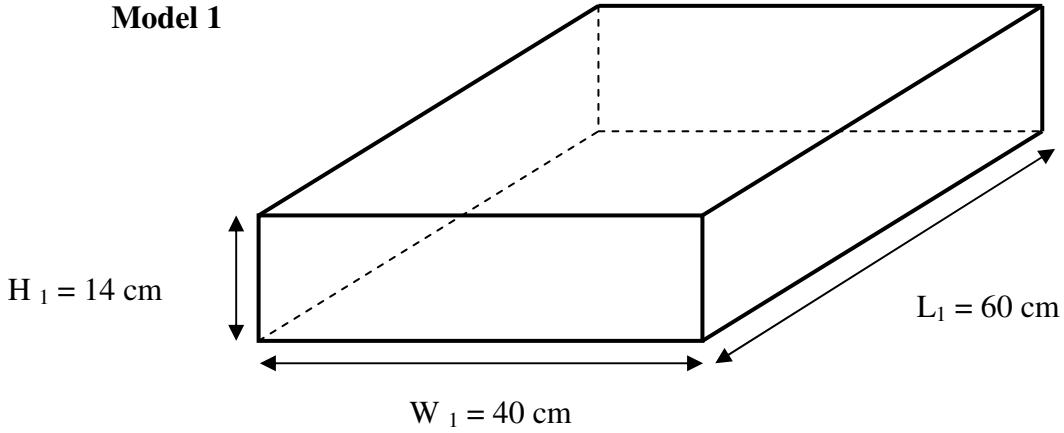
Milk Protein Isolate

Figure 62: Scanning electron micrographs of milk protein isolate. The characteristics shown include: (a), (b), and (c) relatively rough particle surface

5.14. Effect of vacuum packaging on efficiency of using storage space

Efficiency of using storage space was evaluated for the three models presented in Figure 34. The dimensions of the three models and the efficiency of using storage space based on those dimensions were determined as outlined in section 4.7.

Model 1

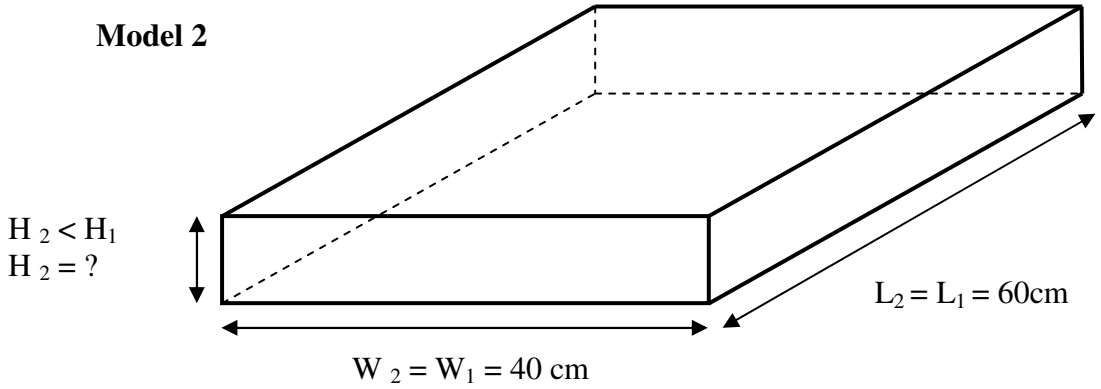


$Weight_1 = 25\ kg = 25000\ g$

$Volume_1 = H_1 \times W_1 \times L_1 = 14 \times 40 \times 60$ **= 33600 cm^3**

$Density_1 = Weight_1 \div Volume_1 = 25000 \div 33600$ **= 0.74 g/cm^3**

Model 2



$Weight_2 = Weight_1 = 25\ kg = 25000\ g$

$Volume_2 = H_2 \times W_2 \times L_2 = (H_2 \times 40 \times 60)\ cm^3$

$Density_2 = Weight_2 \div Volume_2 = 0.88\ g/cm^3 = 25000 \div (H_2 \times 40 \times 60) = 0.88\ g/cm^3$ **$H_2 = 11.84$ cm**

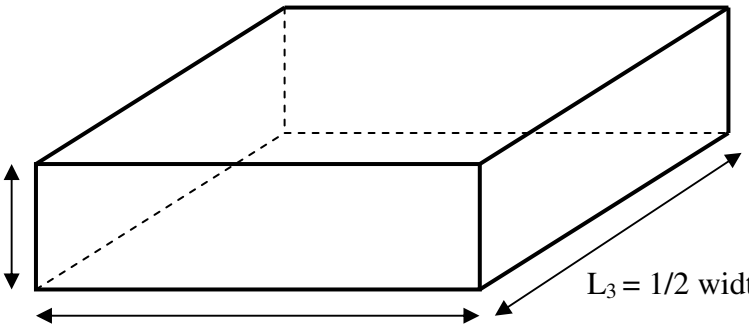
$Volume_2 = H_2 \times W_2 \times L_2 = 11.84 \times 40 \times 60$ **$= 28416\ cm^3$**

Saved space due to vacuum packaging (per bag) = $Volume_1$ □ $Volume_2$ = 33600 □ 28416 **$= 5184\ cm^3 = 316.36\ in^3$**

Saved space due to vacuum packaging (per pallet) = 45 bags per pallet = 45 × 5148 **$= 231660\ cm^3 = 14236.2\ in^3$**

Saved space per bag (percentage) = 100 – [(Volume2 ÷ Volume1) × 100] = 100 - [(28416 ÷ 33600) × 100] = **15.43 %**

Model 3



H_3 = ?

L_3 = 1/2 width of pallet = 101.6/ 2 = 50.8 cm

W_3 = 1/3 length of pallet = 121.9/ 3 = 40.6 cm

$Weight_3 = Weight_3 = 25\ kg = 25000\ g$

$Volume_3 = H_3 \times W_3 \times L_3 = (H_3 \times 40.6 \times 50.8)\ cm3$

$Density_3 = Density_2 = Weight_3 \div Volume_3 = 0.88\ g/cm3$

$= 25000 \div (H_3 \times 40.6 \times 50.8) = 0.88\ g/cm3$ **$H_3 = 13.77$ cm**

$Volume_3 = H_3 \times W_3 \times L_3 = 13.77 \times 40.6 \times 50.8 =$ **28400.3 cm^3**

5.14.1. Calculations

Density

- Density of a 25 kg atmospheric packaged “non fat dry milk” = $Density_1$ = 25000 ÷ 33600 **= 0.74 g/cm3**

As models 2 and 3 are hypothetical models, for the sake of comparison, it was assumed that the density of nonfat dry milk powder in a 25 kg bag is equal to that in a small bag.

- Density of vacuum packaged brick of “non fat dry milk” (small bag) =

Weight of the vacuum packaged “non fat dry milk” ÷ Volume of the vacuum packaged “non fat dry milk”

Weight of the vacuum packaged “non fat dry milk” = 280.35 g

Volume of the vacuum packaged “non fat dry milk” (by water displacement) = 320 cm^3

- Density of vacuum packaged brick of “non fat dry milk” (small brick) = 280.35 ÷ 320 = **0.8762 0.88 g/cm3**

Volume

- Volume1 = H1 × W1 × L1 = 14 × 40 × 60 = **33600 cm3**
- Volume2 = H2 × W2 × L2 = (H2 × 40 × 60) cm3

  Density2 = Weight2 ÷ Volume2 = 0.88 g/cm3

  25000 ÷ (H2 × 40 × 60) = 0.88 g/cm3 H2 = 11.84 cm

- Volume2 = H2 × W2 × L2 = 11.84 × 40 × 60 = **28416 cm3**

Saved space per bag

$$100 – [(\text{Volume2} \div \text{Volume1}) \times 100] =$$

$$100 – [(28416 \div 33600) \times 100] = \mathbf{15\ \%}$$

Vacuum packaging nonfat dry milk resulted in15% space saving per bag.

5.14.2. CAPE Pack analyses

Figure 63 shows CAPE Pack output of model 1 with pallet information, including:

- Dimensions of an individual model 1 bag :
  length = 60 cm = 600 mm / width = 40 cm = 400 mm / height =14 cm =140 mm
- Pallet size = 48 in × 40 in × 6 in = 1219.2 cm × 1016 cm× 152.4 cm
- Load height = height of the piled bags + height of pallet =
  (9 [layers] × 140) + 152.4 = 1412.4 mm

Figure 64 shows CAPE Pack output of model 2 with pallet information, including:

- Dimensions of an individual model 2 bag :
  length = 60 cm = 600 mm / width = 40 cm = 400 mm / height = 11.84 cm = 118.4 mm
- Pallet size = 48 in × 40 in × 6 in = 1219.2 cm × 1016 cm× 152.4 cm
- Load height = height of the piled bags + height of pallet =
  (10 [layers] × 118.4) + 152.4 = 1336.4 mm

Figure 65 shows CAPE Pack output of model 3 with pallet information, including:

- Dimensions of a model 3 bag :

  Length = 50.8 cm = 508 mm / width = 40.6 cm = 406 mm / height =13.77 cm =137.7mm

- Pallet size = 48 in × 40 in × 6 in = 1219.2 cm × 1016 cm× 152.4 cm

- Load height = height of the piled bags + height of pallet =

  (9 [layers] × 137.7) + 152.4 = 1391.7 mm

  Pallet size for the three models were the same and the maximum allowed height of the bags on each pallet was kept constant and equal to the height of the 9 layers of bags in model$_1$: 9 × 140 = 1260 mm (model 1 represented a commercial atmospheric packaged bag and models 2 and 3 represented two prototype vacuum packaged bags with different dimensions but the same density. The weights of models 1, 2, and 3 were equal.)

- Case = an individual bag of powder
- Layer = bags placed at the same level (height) on a pallet form a layer
- Load = a pallet + bags placed on it
- Pallet pattern = the pattern of placing the bags on a pallet (3 ×2 is the common pattern for commercial bags of dairy powder used for models 1 1nd 2) (Bush D., personal communication, Jan.12 2010).

5.14.3. Pallet Efficiency Comparisons, CAPE Output

Model1			
Pallet type	48 × 40	Bags / layer	5
Weight/ pallet	1125	Layers/ load	9
		Bags/ load	45

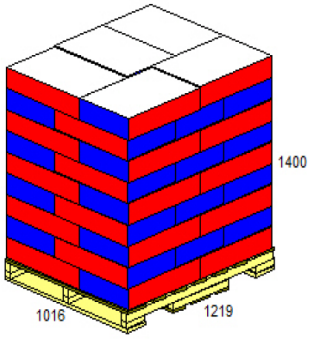


Figure 63: CAPE Pack pallet efficiency output of model$_1$

All the pallet specifications used for model1 are the actual numbers used in dairy industry (Bush D., personal communication, Jan.12 2010).

Model2			
Pallet type	48 × 40	Bags/ layer	5
Weight/ pallet (kg)	1250	Layers/ load	10
Increase in W/P (%)	11	Bags/ load	50

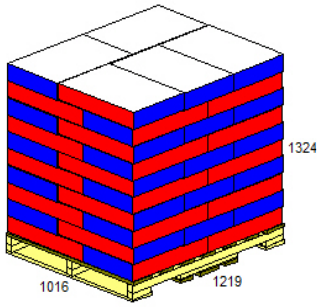


Figure 64: CAPE Pack pallet efficiency output of model$_2$

Model3			
Pallet type	48 × 40	Bags/ layer	6
Weight/ pallet (kg)	1350	Layers/ load	9
Increase in W/P (%)	20	Bags/ load	54

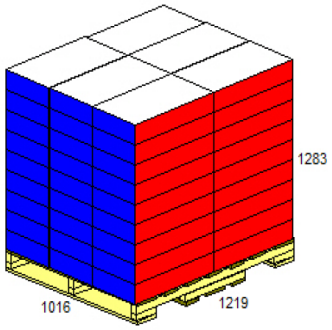


Figure 65: CAPE Pack pallet efficiency output of model$_3$

Vacuum packaging nonfat dry milk, improved the pallet efficiency by 11 and 20 % increased weight per pallet in models 2 and 3, respectively. The weight/ pallet was increased from 1125 kg in model 1 to 1250 kg in model2 and 1350 kg in model 3. The total number of bags per pallet was increased from 45 in model1 to 50 in model 2 and 54 in model3.

5.14.4. Comparison of pallet patterns and efficiency of models 1, 2, and 3

CAPE Pack analyses of pallet patterns and efficiency of models 1, 2, and three revealed that:

1. $Model_1$ had the lowest pallet efficiency, with 5 bags per layer, 9 layers of bags per pallet and overall 45 bags per pallet.
2. $Model_3$ had the highest pallet efficiency, with 6 bags per layer, 9 layers of bags per pallet and overall 54 bags per pallet.
3. $Model_2$ had pallet efficiency between $model_1$ and $model_2$, with 5 bags per layer, 10 layers of bags per pallet and overall 50 bags per pallet.
4. Assuming no consequences due to the additional weight, $model_2$ and $model_3$ allow shipping 5 and 9 additional bags per pallet respectively, for essentially the same cost.
5. The cost effectiveness of the three models are worth quantifying; however, the packaging cost is expected to be the lowest for $model_3$ and the highest for $model_1$, as the cost of packaging per unit of product shipped decreases with increases in the bulk density of the product, i.e., with the mass per unit volume of shipping space taken by product plus its packaging (Brown, 1992).

6. CONCLUSIONS AND LIMITATIONS

The major conclusions of this research are:

1. Vacuum packaging did not have any detrimental effect on the solubility of the powders tested during 12 months storage time.
2. Vacuum packaging improved the color of all the powders tested regardless of the powder type and storage time.
3. There was an age related increase in the particle size, density characteristics, and flowability of the powders tested over 12 months of storage.
4. There were no significant interactions of vacuum pressure and storage time on the physical properties of the powders tested.
5. Vacuum packaging nonfat dry milk resulted in 15% space saving per bag.
6. Vacuum packaging nonfat dry milk, improved the pallet efficiency by 11 and 20 % increased weight per pallet in models 2 and 3, respectively.

The goal of this thesis project was to evaluate the effect of a vacuum packaging method on the quality of dairy powders over a one year storage period, utilizing side gusset bags. Dairy powders packaged under different degrees of vacuum using the side gusset bags showed no significant changes in their solubility and moisture content but some statistically significant changes in their physical properties over the one year storage time. For example, it was observed that particle density, particle size, bulk density, and tapped density of the powders increased over the storage time, while AOR and compressibility decreased over the storage time. In fact, the dairy powders packaged by this method and stored for a longer period had significantly higher particle density, particle size, bulk density, tapped density, and flowabilty but significantly lower

compressibility than the powders that had been packaged by this method but stored for a shorter period.

The powder type had a significant effect on particle density, particle size, bulk density, tapped density, AOR, compressibility, and color values: L-, a-, b-; however, it did not have any significant effect on solubility and moisture content.

It was observed that vacuum pressure had a statistically significant effect on L-, a-, and b-color values of the powders tested, especially whole milk powder. These results are in agreement with previous research showing the same color changes in milk powders, due to oxidation during storage (Nielsen et al., 1997; Stapelfeldt et al., 1997).

Vacuum packaging does increase the efficiency of using the storage space by removing the interstitial air (the air among powder particles) and increasing the density of the powder. For example, storage space calculations for non fat dry milk showed 15.43 % saving in storage space per bag and per pallet, due vacuum packaging.

The significant effect of storage time on physical properties as mentioned above may be explained by a phenomenon called time consolidation which is caused by (1) increased bulk density owing to the powder consolidating over time. (2) Physical and chemical changes occurring during the storage time (Teunou et al., 1999). The increase in bulk density leads to the powder particles being pushed closer together. This results in increased van der Waals interaction leading to greater cohesion, and forming powder particles with higher densities (Bhandari and Hartel, 2005). Other than the increases in particle and bulk density, the same phenomenon is believed to be responsible for the increases in particle size, tapped density, and flowability and also the decrease in compressibility.

The significant impact of powder type on physical properties of the powders can be explained by the differences in composition, particle shape, and the surface morphology of different powders. Using scanning electron microscopy, differences in particle structure and surface morphology of the powders could be observed among different powders. It is known that the protein and total solids contents play a major role in whipping more air into the feed and forming particles of higher porosity and lower density consequentially.

The significant effect of packaging method (vacuum pressure) on L-, a-, and b- color values, especially in higher fat containing powders such as whole milk powder, is likely correlated with the removal of oxygen, preventing lipid oxidation and its consequent color changes in the vacuum packaged powders.

The significant effect of vacuum packaging on storage space is due to an increase in the bulk density of powders caused by removing the interstitial air under vacuum. The saved space is expected to be different for different powders considering the difference in compactability/ compressibility of different powders.

The research findings provide a foundation for future studies investigating the effect of vacuum packaging in side gusset bags on improving efficiency of using storage space and on keeping quality attributes of six types of dairy powders, including the physical properties and solubility, over a long storage time. These insights may assist dairy powder manufacturers in choosing the best packaging method that retains the quality of powders consistently during the storage time while improving the efficiency of using storage space.

7. DIRECTIONS FOR FUTURE RESEARCH

Additional research into the relationship between the effect of storage time and vacuum packaging methods on the quality attributes is needed, to broaden our understanding of both the changes in dairy powders quality and the mechanisms by which removing air from the package and increasing the storage time yields these changes. The future research should also involve the geometrical aspects of the final packaged products to investigate the effect of vacuum packaging on the final cost of dairy powders. Some possible research directions are as follows:

1. Determine the effect of powder source (manufacturing company) on quality attributes of different vacuum packaged dairy powders over the storage time. It must be emphasized that each powder used in the current study had been obtained from only a single manufacturer. Therefore, all the significant differences in powder quality attributed to the effect of powder type are well worth validating by obtaining each type of powders from different manufacturers.
2. Investigate the effect of vacuum packaging and storage time on the sensory attributes and flavor profile of dairy powders. The differences observed in L-, a-, and b- color values between atmospheric and vacuum packaged whole milk powder are a good indicator of the likely effect of vacuum packaging on lipid oxidation which is worth further investigating, by methods measuring lipid oxidation products, through methods such as peroxide value analysis (Hahm and Min, 1995; Ulberth and Roubicek, 1995; Jacobsen, 1999; Ruiz et al., 2001; van der Merwe et al., 2003; Chung et al., 2004).

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Appendix A: Data for Compositional Properties

Table 16: Compositional properties of the powders tested right after receiving from the munfacturing companies. Means and standard deviations were calculated from two measurements.

Product	Protein %	Fat %	Ash %	Lactose %
NFDM	36.16 ± 0.21	0.72 ± 0.12	7.90 ± 0.01	52.34 ± 0.24
BMP	35.13 ± 0.37	5.31 ± 0.09	7.11 ± 0.02	49.56 ± 0.15
MPI	86.62 ± 0.66	1.50 ± 0.03	5.23 ± 0.01	1.22 ± 0.08
WPC80	78.34 ± 0.75	6.55 ± 0.11	3.54 ± 0.01	5.50 ± 0.17
WMP	26.87 ± 0.24	25.37 ± 0.23	5.83 ± 0.05	37.65 ± 0.06
SWP	13.06 ± 0.18	1.42 ± 0.06	8.33 ± 0.01	73.55 ± 0.25

Appendix B: Minitab Output for Particle Density

General Linear Model: Particle density versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for Particle density, using Adjusted SS for Tests

Source             DF     Seq SS     Adj SS     Adj MS      F      P
Time                2  0.0042321  0.0042321  0.0021161  77.19  0.000
Powder              5  0.0021047  0.0021047  0.0004209  15.35  0.000
Vac. Press.         3  0.0000128  0.0000128  0.0000043   0.16  0.926
Time*Vac. Press.    6  0.0001093  0.0001093  0.0000182   0.66  0.679
Error             127  0.0034816  0.0034816  0.0000274
Total             143  0.0099404


S = 0.00523585   R-Sq = 64.98%   R-Sq(adj) = 60.56%


Unusual Observations for Particle density

     Particle
Obs   density       Fit    SE Fit   Residual  St Resid
 36  0.004900  0.014833  0.001799  -0.009933     -2.02 R
 46  0.035400  0.022500  0.001799   0.012900      2.62 R
 48  0.032600  0.021592  0.001799   0.011008      2.24 R
 57  0.028400  0.018317  0.001799   0.010083      2.05 R
 90  0.033300  0.022852  0.001799   0.010448      2.12 R
121  0.003800  0.013917  0.001799  -0.010117     -2.06 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
12    48   0.0  A
 6    48   0.0    B
 3    48   0.0      C

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
50vacpet     36   0.0  A
99vacpet     36   0.0  A
25vacpet     36   0.0  A
0vacpet      36   0.0  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence
```

Time	Vac. Press.	N	Mean	Grouping
12	99vacpet	12	0.0	A
12	50vacpet	12	0.0	A
12	25vacpet	12	0.0	A
12	0vacpet	12	0.0	A B
6	50vacpet	12	0.0	B C
6	25vacpet	12	0.0	B C
6	0vacpet	12	0.0	C
6	99vacpet	12	0.0	C
3	25vacpet	12	0.0	C
3	99vacpet	12	0.0	C
3	0vacpet	12	0.0	C
3	50vacpet	12	0.0	C

Means that do not share a letter are significantly different.

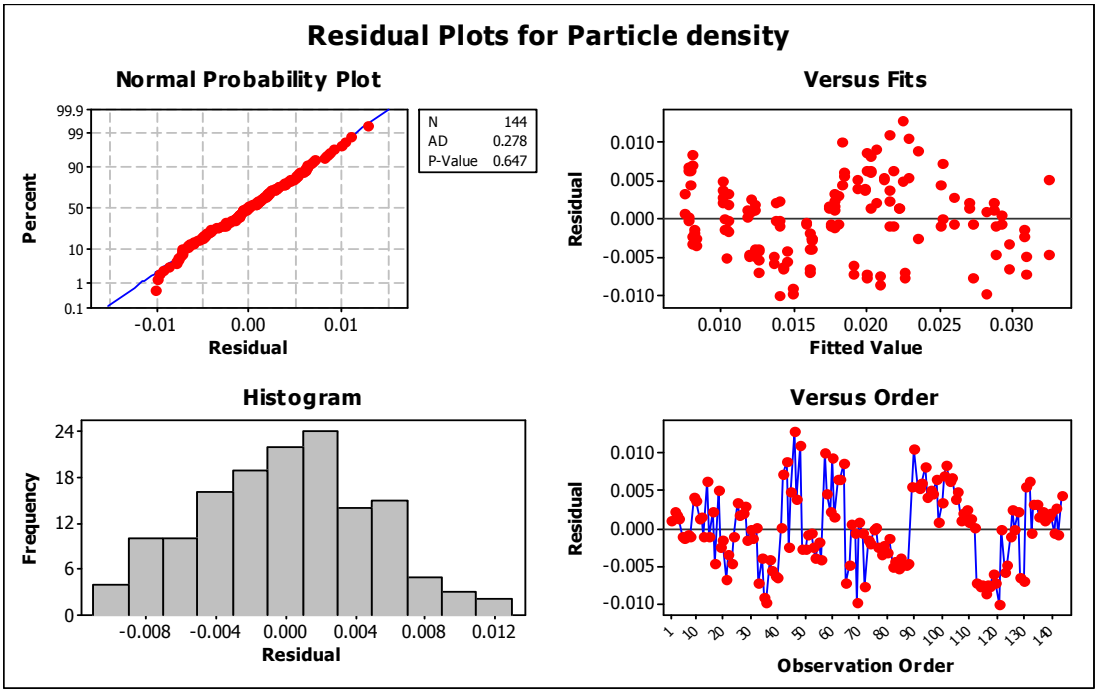

Figure 66: Residual plots for particle density of powders as determined by Accupyc 1340 pycnometer for 3, 6 and 12 month storage; Top Left: Normal probability plot (p = 0.647); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix C: Minitab Output for Particle Size

General Linear Model: Particle size versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for Particle size, using Adjusted SS for Tests

Source             DF   Seq SS   Adj SS  Adj MS      F      P
Time                2   503.81   503.81  251.90  68.10  0.000
Powder              5  1447.03  1447.03  289.41  78.23  0.000
Vac. Press.         3     1.19     1.19    0.40   0.11  0.956
Time*Vac. Press.    6     2.51     2.51    0.42   0.11  0.995
Error             127   469.81   469.81    3.70
Total             143  2424.34


S = 1.92336   R-Sq = 80.62%   R-Sq(adj) = 78.18%


Unusual Observations for Particle size

     Particle
Obs      size      Fit  SE Fit  Residual  St Resid
 79    3.4000  -0.4878  0.6608    3.8878      2.15 R
121   -1.3100   2.3443  0.6608   -3.6543     -2.02 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
12    48   7.6  A
 6    48   4.9    B
 3    48   3.0      C

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
0vacpet      36   5.2  A
50vacpet     36   5.2  A
25vacpet     36   5.2  A
99vacpet     36   5.0  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Time  Vac. Press.   N  Mean  Grouping
12    0vacpet      12   7.8  A
12    50vacpet     12   7.7  A B
```

12	25vacpet	12	7.5	A B C
12	99vacpet	12	7.3	A B C D
6	25vacpet	12	5.1	B C D E
6	99vacpet	12	4.9	C D E
6	0vacpet	12	4.8	D E
6	50vacpet	12	4.8	D E
3	50vacpet	12	3.2	E
3	0vacpet	12	3.1	E
3	25vacpet	12	2.9	E
3	99vacpet	12	2.8	E

Means that do not share a letter are significantly different.

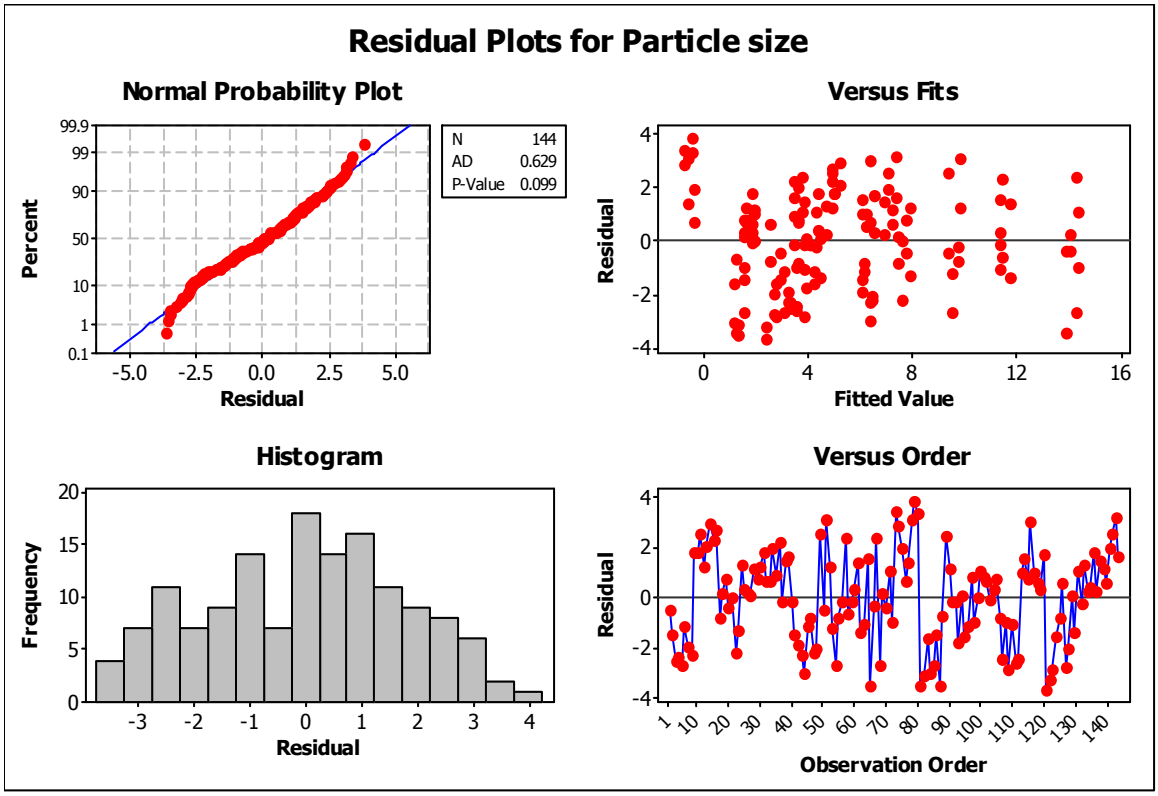

Figure 67: Residual plots for mean particle size of powders as determined by Coulter LS 230; Top Left: Normal probability plot (p = 0.099); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix D: Minitab Output for Bulk Density

General Linear Model: Bulk density versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for Bulk density, using Adjusted SS for Tests

Source             DF     Seq SS     Adj SS     Adj MS      F      P
Time                2  0.0077215  0.0077215  0.0038607  40.21  0.000
Powder              5  0.0186022  0.0186022  0.0037204  38.74  0.000
Vac. Press.         3  0.0000488  0.0000488  0.0000163   0.17  0.917
Time*Vac. Press.    6  0.0003225  0.0003225  0.0000537   0.56  0.762
Error             127  0.0121950  0.0121950  0.0000960
Total             143  0.0388899


S = 0.00979918   R-Sq = 68.64%   R-Sq(adj) = 64.69%


Unusual Observations for Bulk density

Obs  Bulk density       Fit    SE Fit   Residual  St Resid
100      0.042600  0.022252  0.003367   0.020348      2.21 R
118      0.016800  0.035402  0.003367  -0.018602     -2.02 R
125      0.000000  0.020698  0.003367  -0.020698     -2.25 R
128     -0.010000  0.017246  0.003367  -0.027246     -2.96 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
12    48   0.0  A
 6    48   0.0  A
 3    48   0.0    B

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
99vacpet     36   0.0  A
50vacpet     36   0.0  A
25vacpet     36   0.0  A
0vacpet      36   0.0  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Time  Vac. Press.   N  Mean  Grouping
12    99vacpet     12   0.0  A
 6    99vacpet     12   0.0  A
```

12	25vacpet	12	0.0	A B
6	50vacpet	12	0.0	A B
6	0vacpet	12	0.0	A B
12	0vacpet	12	0.0	A B
12	50vacpet	12	0.0	A B C
6	25vacpet	12	0.0	A B C
3	50vacpet	12	0.0	B C D
3	25vacpet	12	0.0	C D
3	0vacpet	12	0.0	D
3	99vacpet	12	0.0	D

Means that do not share a letter are significantly different

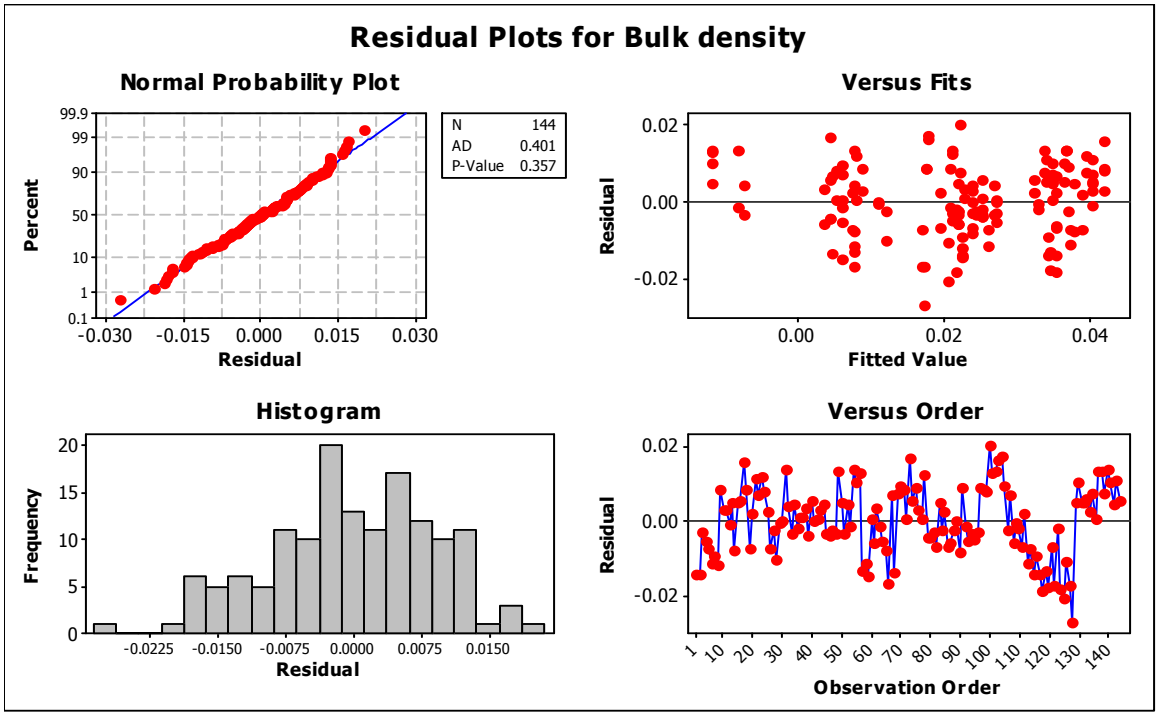

Figure 68: Residual plots for bulk density of powders as determined by IDF standard metod: 134A,1995; Top Left: Normal probability plot (p = 0.357); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix E: Minitab Output for Tapped Density

General Linear Model: Tapped denity versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for Tapped denity, using Adjusted SS for Tests

Source             DF     Seq SS     Adj SS     Adj MS      F      P
Time                2  0.0054121  0.0054121  0.0027061  23.62  0.000
Powder              5  0.0287631  0.0287631  0.0057526  50.22  0.000
Vac. Press.         3  0.0003998  0.0003998  0.0001333   1.16  0.326
Time*Vac. Press.    6  0.0004880  0.0004880  0.0000813   0.71  0.642
Error             127  0.0145475  0.0145475  0.0001145
Total             143  0.0496105


S = 0.0107027   R-Sq = 70.68%   R-Sq(adj) = 66.98%


Unusual Observations for Tapped denity

Obs  Tapped denity       Fit    SE Fit   Residual  St Resid
  2       0.014103  0.038199  0.003677  -0.024097     -2.40 R
  4       0.014329  0.034670  0.003677  -0.020341     -2.02 R
  5       0.012961  0.035053  0.003677  -0.022092     -2.20 R
  7       0.013141  0.034479  0.003677  -0.021338     -2.12 R
  8       0.010483  0.034479  0.003677  -0.023996     -2.39 R
  9       0.022785  0.044350  0.003677  -0.021565     -2.15 R
 10       0.021443  0.044350  0.003677  -0.022906     -2.28 R
 14       0.066364  0.045049  0.003677   0.021315      2.12 R
 17       0.074684  0.051950  0.003677   0.022734      2.26 R
 20       0.074722  0.053038  0.003677   0.021685      2.16 R
 23       0.067179  0.044725  0.003677   0.022454      2.23 R
 48      -0.010000  0.011576  0.003677  -0.021576     -2.15 R
119      -0.010000  0.010883  0.003677  -0.020883     -2.08 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
12    48   0.0  A
 6    48   0.0  A
 3    48   0.0    B

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
50vacpet     36   0.0  A
99vacpet     36   0.0  A
25vacpet     36   0.0  A
0vacpet      36   0.0  A
```

Means that do not share a letter are significantly different.

Grouping Information Using Tukey Method and 95.0% Confidence

Time	Vac. Press.	N	Mean	Grouping
12	50vacpet	12	0.0	A
12	99vacpet	12	0.0	A B
6	50vacpet	12	0.0	A B
12	25vacpet	12	0.0	A B
6	0vacpet	12	0.0	A B C
6	25vacpet	12	0.0	A B C
12	0vacpet	12	0.0	A B C
6	99vacpet	12	0.0	A B C
3	99vacpet	12	0.0	B C
3	25vacpet	12	0.0	C
3	50vacpet	12	0.0	C
3	0vacpet	12	0.0	C

Means that do not share a letter are significantly different.

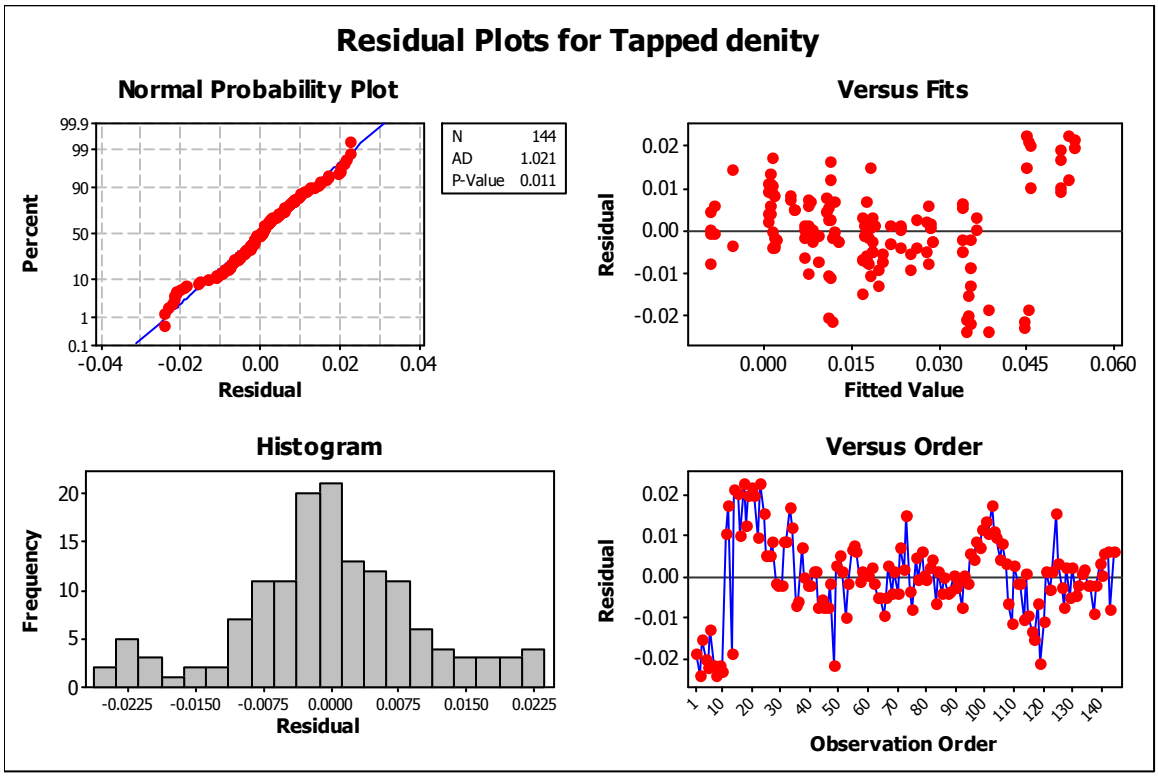

Figure 69: Residual plots for tapped density of powders as determined by IDF standard method 134A:1995; Top Left: Normal probability plot (p = 0.011); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix F: Minitab Output for Angle of Repose

General Linear Model: AOR versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for AOR, using Adjusted SS for Tests

Source             DF   Seq SS   Adj SS  Adj MS      F      P
Time                2   38.741   38.741  19.370  16.53  0.000
Powder              5  154.116  154.116  30.823  26.30  0.000
Vac. Press.         3    0.219    0.219   0.073   0.06  0.980
Time*Vac. Press.    6    4.776    4.776   0.796   0.68  0.667
Error             127  148.857  148.857   1.172
Total             143  346.710


S = 1.08264   R-Sq = 57.07%   R-Sq(adj) = 51.66%


Unusual Observations for AOR

Obs       AOR       Fit   SE Fit  Residual  St Resid
 14  -3.22000  -1.05486  0.37199  -2.16514     -2.13 R
 31  -0.17000  -2.30611  0.37199   2.13611      2.10 R
 37   0.24000  -2.36861  0.37199   2.60861      2.57 R
 42  -5.18000  -2.99444  0.37199  -2.18556     -2.15 R
 43  -6.24000  -3.54694  0.37199  -2.69306     -2.65 R
 47  -5.90000  -3.28361  0.37199  -2.61639     -2.57 R
 51  -5.06000  -2.76569  0.37199  -2.29431     -2.26 R
113  -1.90000  -4.09236  0.37199   2.19236      2.16 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
 6    48  -1.8  A
 3    48  -2.0  A
12    48  -3.0     B

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
99vacpet     36  -2.2  A
0vacpet      36  -2.3  A
25vacpet     36  -2.3  A
50vacpet     36  -2.3  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence
```

Time	Vac. Press.	N	Mean	Grouping
6	50vacpet	12	-1.5	A
6	0vacpet	12	-1.7	A B
3	25vacpet	12	-1.8	A B
6	99vacpet	12	-1.8	A B
3	0vacpet	12	-2.0	A B C
6	25vacpet	12	-2.1	A B C
3	99vacpet	12	-2.1	A B C
3	50vacpet	12	-2.2	A B C
12	99vacpet	12	-2.7	A B C
12	25vacpet	12	-2.9	A B C
12	0vacpet	12	-3.0	B C
12	50vacpet	12	-3.3	C

Means that do not share a letter are significantly different.

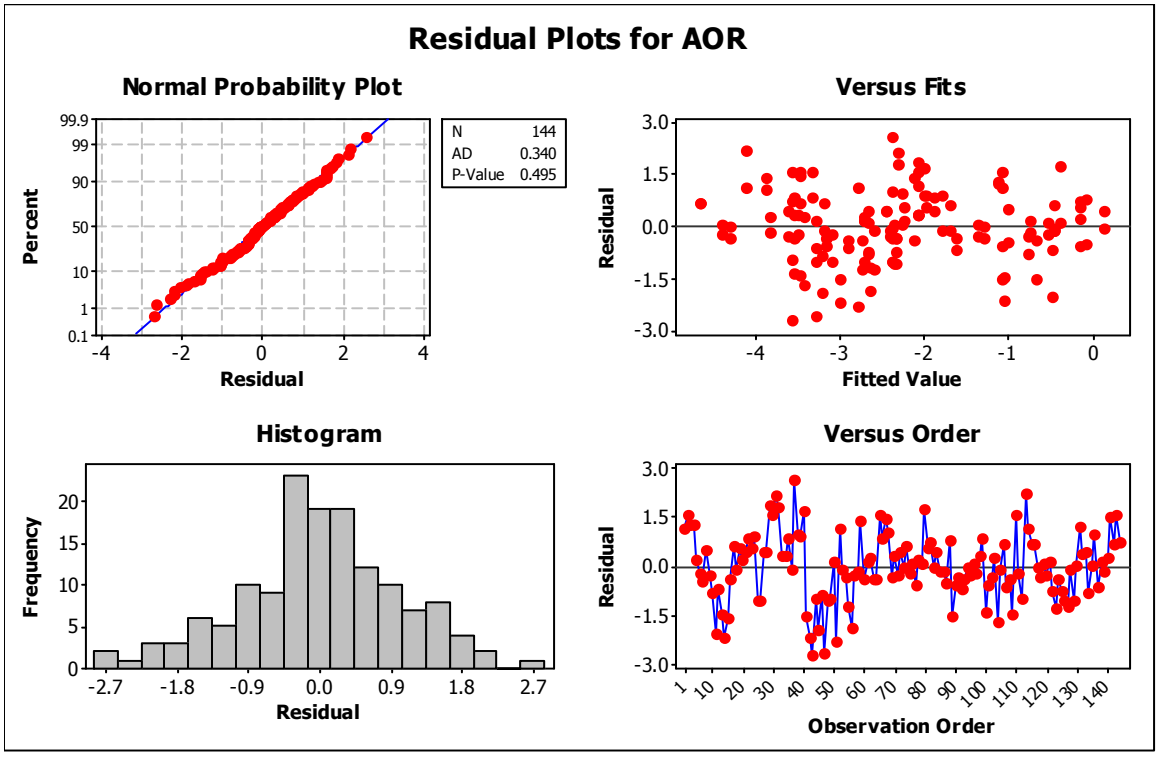

Figure 70: Residual plots for angle of repose (AOR) of powders as determined by Mark 4 Angle of Repose Tester; Top Left: Normal probability plot (p = 0.495); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix G: Minitab Output for Compressibility

Minitab Output Compressibility

General Linear Model: Compressibility versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for Compressibility, using Adjusted SS for Tests

Source             DF    Seq SS   Adj SS   Adj MS      F      P
Time                2    2.9771   2.9771   1.4885   5.68  0.004
Powder              5   70.5971  70.5971  14.1194  53.90  0.000
Vac. Press.         3    0.0638   0.0638   0.0213   0.08  0.970
Time*Vac. Press.    6    2.8634   2.8634   0.4772   1.82  0.100
Error             127   33.2668  33.2668   0.2619
Total             143  109.7682


S = 0.511804   R-Sq = 69.69%   R-Sq(adj) = 65.88%


Unusual Observations for Compressibility

Obs  Compressibility       Fit   SE Fit  Residual  St Resid
 38          1.61250   0.31525  0.17585   1.29725      2.70 R
 47          1.21700   0.21030  0.17585   1.00670      2.09 R
 55         -2.28100  -1.28085  0.17585  -1.00015     -2.08 R
125          0.32450  -0.97450  0.17585   1.29900      2.70 R
128          0.91700  -0.76491  0.17585   1.68191      3.50 R
142         -2.59600  -1.05558  0.17585  -1.54042     -3.20 R
144         -2.38900  -1.19537  0.17585  -1.19363     -2.48 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
 3    48  -0.4  A
 6    48  -0.6    B
12    48  -0.7    B

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
50vacpet     36  -0.5  A
99vacpet     36  -0.5  A
25vacpet     36  -0.6  A
0vacpet      36  -0.6  A

Means that do not share a letter are significantly different.
```

Grouping Information Using Tukey Method and 95.0% Confidence

Time	Vac. Press.	N	Mean	Grouping
3	50vacpet	12	-0.1	A
3	0vacpet	12	-0.3	A B
12	99vacpet	12	-0.5	A B
3	25vacpet	12	-0.5	A B
3	99vacpet	12	-0.5	A B
6	50vacpet	12	-0.5	A B
12	25vacpet	12	-0.6	A B
6	25vacpet	12	-0.6	A B
6	99vacpet	12	-0.6	A B
12	0vacpet	12	-0.7	A B
6	0vacpet	12	-0.8	A B
12	50vacpet	12	-0.9	B

Means that do not share a letter are significantly different.

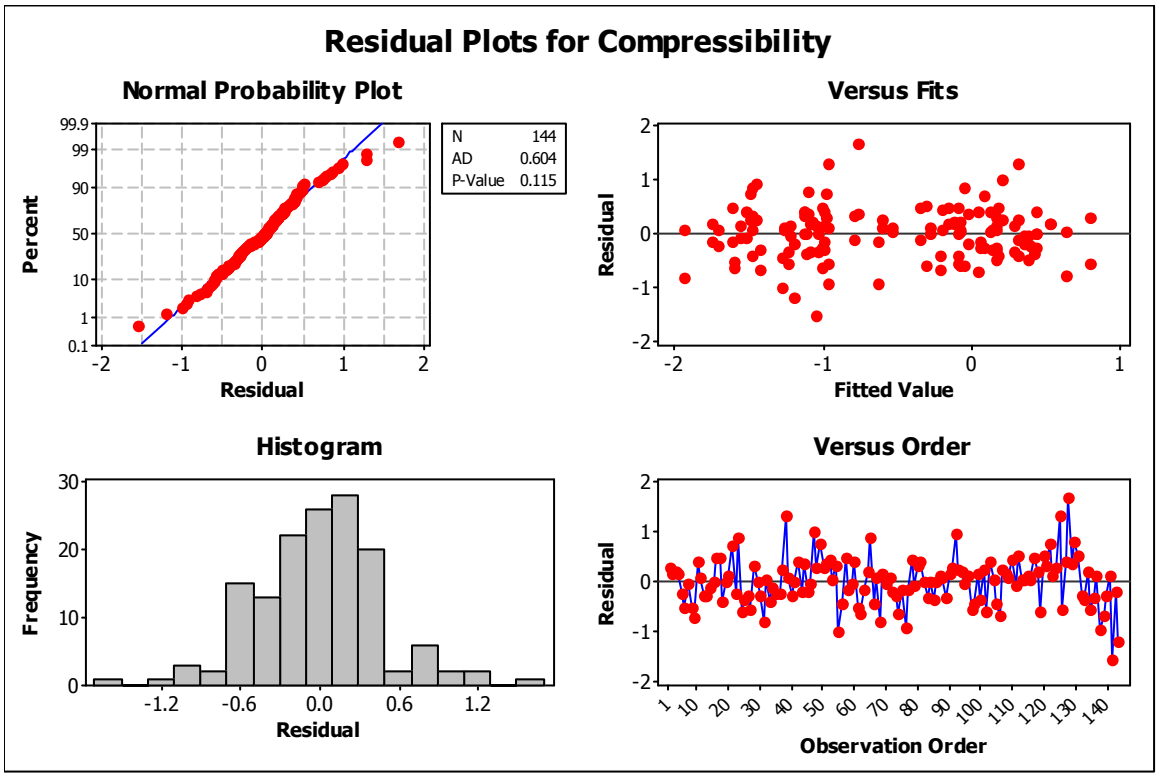

Figure 71: Residual plots for compressibility of powders as determined by TA-XT2 Texture Analyzer using a back extrusion A/BE assembly; Top Left: Normal probability plot (p = 0.115); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix H: Minitab Output for L-value

General Linear Model: L versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for L, using Adjusted SS for Tests

Source             DF   Seq SS   Adj SS  Adj MS     F      P
Time                2   1.1726   1.1726  0.5863  4.04  0.020
Powder              5   6.8016   6.8016  1.3603  9.38  0.000
Vac. Press.         3   2.1605   2.1605  0.7202  4.97  0.003
Time*Vac. Press.    6   0.1003   0.1003  0.0167  0.12  0.994
Error             127  18.4187  18.4187  0.1450
Total             143  28.6537


S = 0.380827   R-Sq = 35.72%   R-Sq(adj) = 27.62%


Unusual Observations for L

Obs         L       Fit   SE Fit  Residual  St Resid
  9  -1.25000  -0.12507  0.13085  -1.12493     -3.15 R
 10   0.66000  -0.12507  0.13085   0.78507      2.20 R
 24  -1.42000  -0.54924  0.13085  -0.87076     -2.43 R
 31   0.65000  -0.13674  0.13085   0.78674      2.20 R
 44  -0.90000  -0.17174  0.13085  -0.72826     -2.04 R
 72  -2.30000  -1.05965  0.13085  -1.24035     -3.47 R
 76   0.89000   0.00035  0.13085   0.88965      2.49 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
 3    48  -0.2  A
 6    48  -0.3  A B
12    48  -0.4    B

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
25vacpet     36  -0.2  A
99vacpet     36  -0.2  A
50vacpet     36  -0.3  A B
0vacpet      36  -0.5    B

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence
```

Time	Vac. Press.	N	Mean	Grouping
3	25vacpet	12	-0.1	A
3	99vacpet	12	-0.1	A
3	50vacpet	12	-0.1	A B
6	99vacpet	12	-0.2	A B
6	25vacpet	12	-0.2	A B
12	99vacpet	12	-0.3	A B
12	25vacpet	12	-0.3	A B
6	50vacpet	12	-0.3	A B
3	0vacpet	12	-0.3	A B
12	50vacpet	12	-0.3	A B
6	0vacpet	12	-0.5	A B
12	0vacpet	12	-0.6	B

Means that do not share a letter are significantly different.

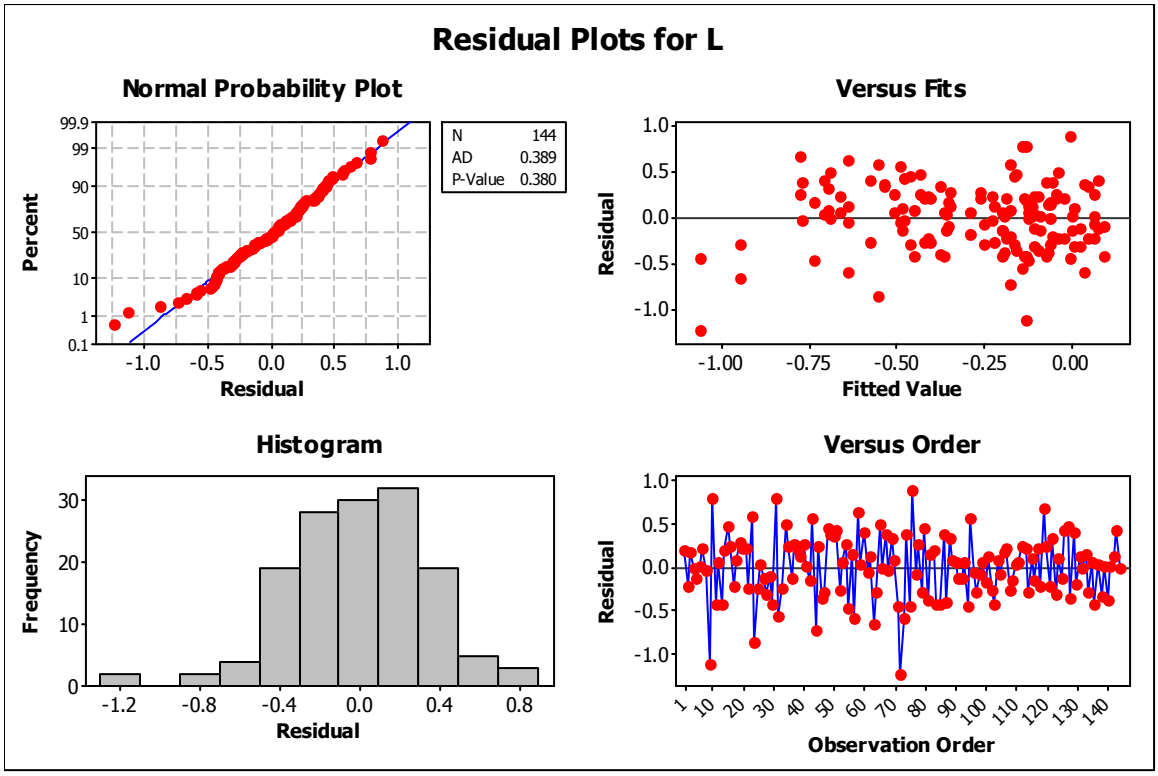

Figure 72: Residual plots for Hunter's L- color value of powders as determined by Ultra Scan XE Spectrophotometer; Top Left: Normal probability plot (p = 0.380); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix I: Minitab Output for a-value

General Linear Model: a versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for a, using Adjusted SS for Tests

Source             DF    Seq SS    Adj SS    Adj MS      F      P
Time                2  0.003501  0.003501  0.001751   1.38  0.256
Powder              5  0.425212  0.425212  0.085042  66.98  0.000
Vac. Press.         3  0.017302  0.017302  0.005767   4.54  0.005
Time*Vac. Press.    6  0.004421  0.004421  0.000737   0.58  0.745
Error             127  0.161247  0.161247  0.001270
Total             143  0.611683


S = 0.0356323   R-Sq = 73.64%   R-Sq(adj) = 70.32%


Unusual Observations for a

Obs          a        Fit    SE Fit   Residual  St Resid
  8   0.200000   0.028403  0.012243   0.171597      5.13 R
 17  -0.080000  -0.012431  0.012243  -0.067569     -2.02 R
 21  -0.080000  -0.012431  0.012243  -0.067569     -2.02 R
 42   0.180000   0.108819  0.012243   0.071181      2.13 R
 47   0.040000   0.125486  0.012243  -0.085486     -2.55 R
 71   0.090000   0.017986  0.012243   0.072014      2.15 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
 3    48   0.0  A
 6    48   0.0  A
12    48  -0.0  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
0vacpet      36   0.0  A
25vacpet     36  -0.0    B
99vacpet     36  -0.0    B
50vacpet     36  -0.0    B

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Time  Vac. Press.   N  Mean  Grouping
```

```
 3     0vacpet       12    0.0  A
 6     0vacpet       12    0.0  A B
12     0vacpet       12    0.0  A B
 6     50vacpet      12    0.0  A B
 3     99vacpet      12    0.0  A B
 3     50vacpet      12    0.0  A B
 3     25vacpet      12    0.0  A B
12     99vacpet      12   -0.0  A B
12     25vacpet      12   -0.0  A B
 6     25vacpet      12   -0.0  A B
 6     99vacpet      12   -0.0  A B
12     50vacpet      12   -0.0     B

Means that do not share a letter are significantly different.
```

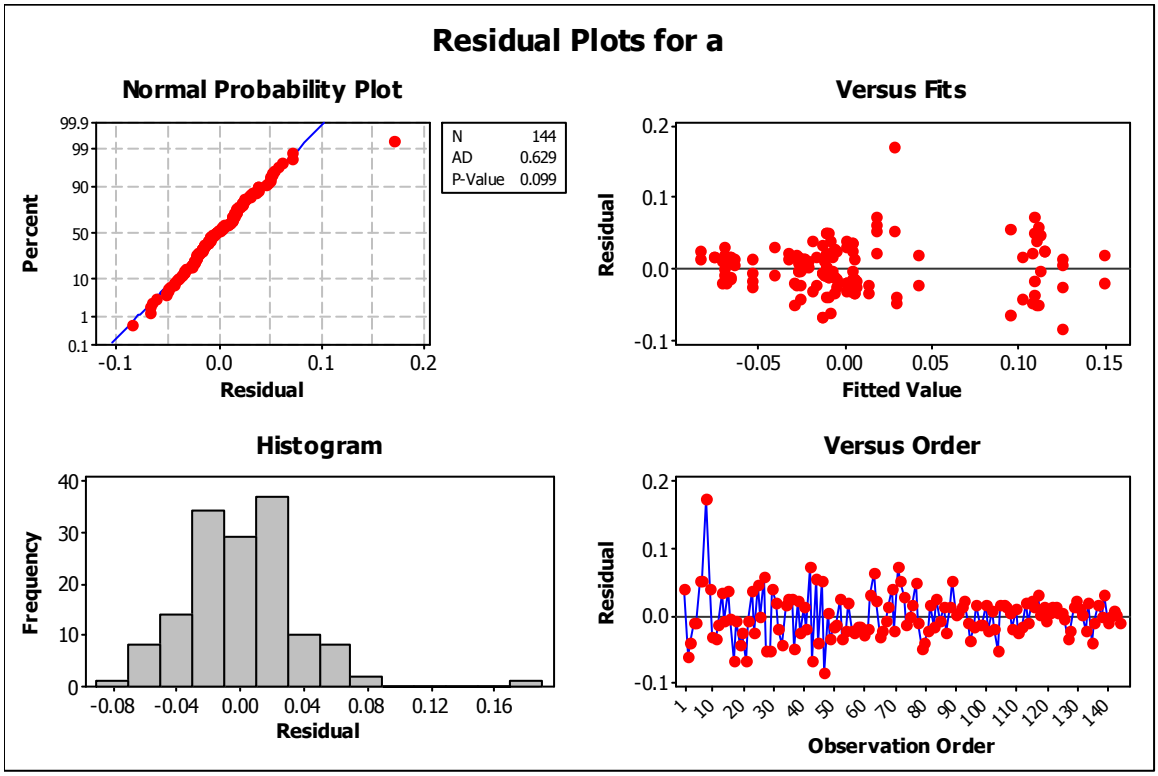

Figure 73: Residual plots for Hunter's a- color value of powders as determined by Ultra Scan XE Spectrophotometer; Top Left: Normal probability plot (p = 0.099); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix J: Minitab Output for b-value

General Linear Model: b versus Time, Powder, Vac. Press.

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for b, using Adjusted SS for Tests

Source             DF    Seq SS   Adj SS   Adj MS     F      P
Time                2   0.04651  0.04651  0.02325  0.37  0.689
Powder              5   1.05998  1.05998  0.21200  3.41  0.006
Vac. Press.         3   1.12420  1.12420  0.37473  6.02  0.001
Time*Vac. Press.    6   0.21483  0.21483  0.03581  0.58  0.749
Error             127   7.90475  7.90475  0.06224
Total             143  10.35027


S = 0.249484   R-Sq = 23.63%   R-Sq(adj) = 14.01%


Unusual Observations for b

Obs          b        Fit    SE Fit   Residual  St Resid
 34   0.420000  -0.049653  0.085721   0.469653      2.00 R
 40  -0.310000   0.237014  0.085721  -0.547014     -2.33 R
 97  -0.750000  -0.210903  0.085721  -0.539097     -2.30 R
 99  -0.810000  -0.188403  0.085721  -0.621597     -2.65 R
100   0.400000  -0.188403  0.085721   0.588403      2.51 R
113   0.790000  -0.156736  0.085721   0.946736      4.04 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
12    48  -0.0  A
 6    48  -0.0  A
 3    48  -0.1  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
0vacpet      36   0.1  A
25vacpet     36  -0.1    B
50vacpet     36  -0.1    B
99vacpet     36  -0.1    B

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Time  Vac. Press.   N  Mean  Grouping
```

```
12     0vacpet       12    0.2  A
 6     0vacpet       12    0.2  A
 3     0vacpet       12    0.0  A
 3     25vacpet      12   -0.0  A
12     99vacpet      12   -0.1  A
 6     25vacpet      12   -0.1  A
 6     50vacpet      12   -0.1  A
 3     50vacpet      12   -0.1  A
12     25vacpet      12   -0.1  A
12     50vacpet      12   -0.1  A
 3     99vacpet      12   -0.1  A
 6     99vacpet      12   -0.1  A

Means that do not share a letter are significantly different.
```

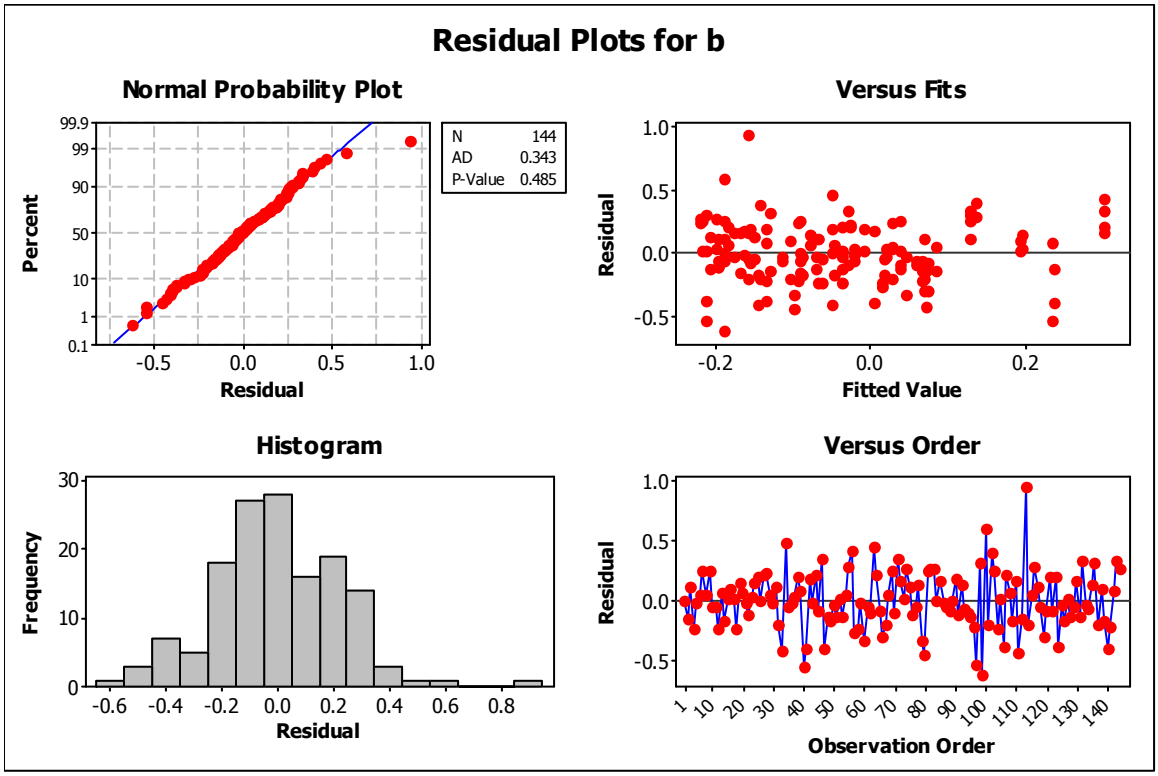

Figure 74: Residual plots for Hunter's b- color value of powders as determined by Ultra Scan XE Spectrophotometer; Top Left: Normal probability plot (p = 0.485); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order

Appendix $K_{(1\&2)}$: Minitab Output for Moisture

1.General Linear Model: Moisture versus Time, Powder, Vac. Press. (Including "0 time" data)

```
Factor       Type    Levels  Values
Time         fixed        3  3, 6, 12
Powder       random       6  BM, MPI, NFDM, SW, WMP, WPC
Vac. Press.  fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for Moisture, using Adjusted SS for Tests

Source             DF   Seq SS   Adj SS   Adj MS     F      P
Time                2  0.00644  0.00644  0.00322  0.17  0.847
Powder              5  0.21921  0.21921  0.04384  2.27  0.052
Vac. Press.         3  0.01027  0.01027  0.00342  0.18  0.912
Time*Vac. Press.    6  0.04634  0.04634  0.00772  0.40  0.878
Error             127  2.45479  2.45479  0.01933
Total             143  2.73706


S = 0.139029   R-Sq = 10.31%   R-Sq(adj) = 0.00%


Unusual Observations for Moisture

Obs  Moisture        Fit    SE Fit  Residual  St Resid
 62  0.140000  -0.122361  0.047769  0.262361      2.01 R
103  0.210000  -0.100694  0.047769  0.310694      2.38 R
107  0.190000  -0.111528  0.047769  0.301528      2.31 R
127  0.180000  -0.105278  0.047769  0.285278      2.18 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time   N  Mean  Grouping
12    48  -0.1  A
 6    48  -0.1  A
 3    48  -0.1  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Vac. Press.   N  Mean  Grouping
25vacpet     36  -0.1  A
0vacpet      36  -0.1  A
50vacpet     36  -0.1  A
99vacpet     36  -0.1  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Time  Vac. Press.   N  Mean  Grouping
```

```
12     25vacpet      12   -0.1   A
 6     25vacpet      12   -0.1   A
 3     50vacpet      12   -0.1   A
 6     0vacpet       12   -0.1   A
 3     0vacpet       12   -0.1   A
12     0vacpet       12   -0.1   A
 6     50vacpet      12   -0.1   A
 6     99vacpet      12   -0.1   A
12     99vacpet      12   -0.1   A
12     50vacpet      12   -0.1   A
 3     99vacpet      12   -0.1   A
 3     25vacpet      12   -0.2   A

Means that do not share a letter are significantly different.
```

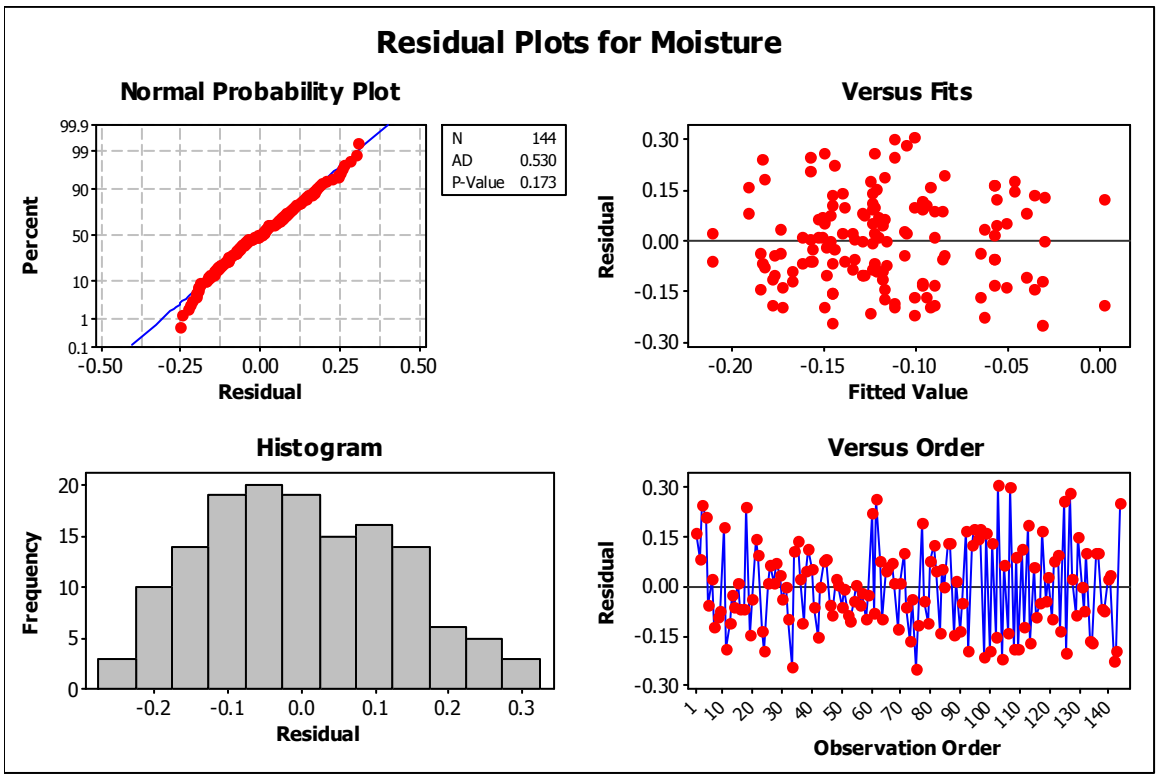

Figure 75: Residual plots for moisture content of powders as determined by drying for 5 h in a vacuum oven at 100ºC; Top Left: Normal probability plot (p = 0.173); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order (Including " 0 time" data)

2. General Linear Model: Moisture versus Time (mo), Powder, Pack-co (Excluding "0 time" data)

```
Factor     Type    Levels  Values
Time (mo)  fixed        3  3, 6, 12
Powder     random       6  BM, MPI, NFDM, SW, WMP, WPC
Pack-co    fixed        4  0vacpet, 25vacpet, 50vacpet, 99vacpet


Analysis of Variance for Moisture, using Adjusted SS for Tests

Source              DF    Seq SS    Adj SS   Adj MS        F      P
Time (mo)            2    0.0064    0.0064   0.0032     0.17  0.847
Powder               5  116.0692  116.0692  23.2138  1200.98  0.000
Pack-co              3    0.0103    0.0103   0.0034     0.18  0.912
Time (mo)*Pack-co    6    0.0463    0.0463   0.0077     0.40  0.878
Error              127    2.4548    2.4548   0.0193
Total              143  118.5871


S = 0.139029   R-Sq = 97.93%   R-Sq(adj) = 97.67%


Unusual Observations for Moisture

Obs  Moisture      Fit   SE Fit  Residual  St Resid
 62   4.43000  4.16764  0.04777   0.26236      2.01 R
103   3.89000  3.57931  0.04777   0.31069      2.38 R
107   3.87000  3.56847  0.04777   0.30153      2.31 R
127   5.65000  5.36472  0.04777   0.28528      2.18 R

R denotes an observation with a large standardized residual.


Grouping Information Using Tukey Method and 95.0% Confidence

Time
(mo)   N  Mean  Grouping
12    48   4.2  A
 6    48   4.2  A
 3    48   4.2  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Pack-co    N  Mean  Grouping
25vacpet  36   4.2  A
0vacpet   36   4.2  A
50vacpet  36   4.2  A
99vacpet  36   4.2  A

Means that do not share a letter are significantly different.


Grouping Information Using Tukey Method and 95.0% Confidence

Time
(mo)  Pack-co    N  Mean  Grouping
12    25vacpet  12   4.2  A
```

```
 6     25vacpet   12    4.2   A
 3     50vacpet   12    4.2   A
 6     0vacpet    12    4.2   A
 3     0vacpet    12    4.2   A
12     0vacpet    12    4.2   A
 6     50vacpet   12    4.2   A
 6     99vacpet   12    4.2   A
12     99vacpet   12    4.2   A
12     50vacpet   12    4.2   A
 3     99vacpet   12    4.1   A
 3     25vacpet   12    4.1   A

Means that do not share a letter are significantly different.
```

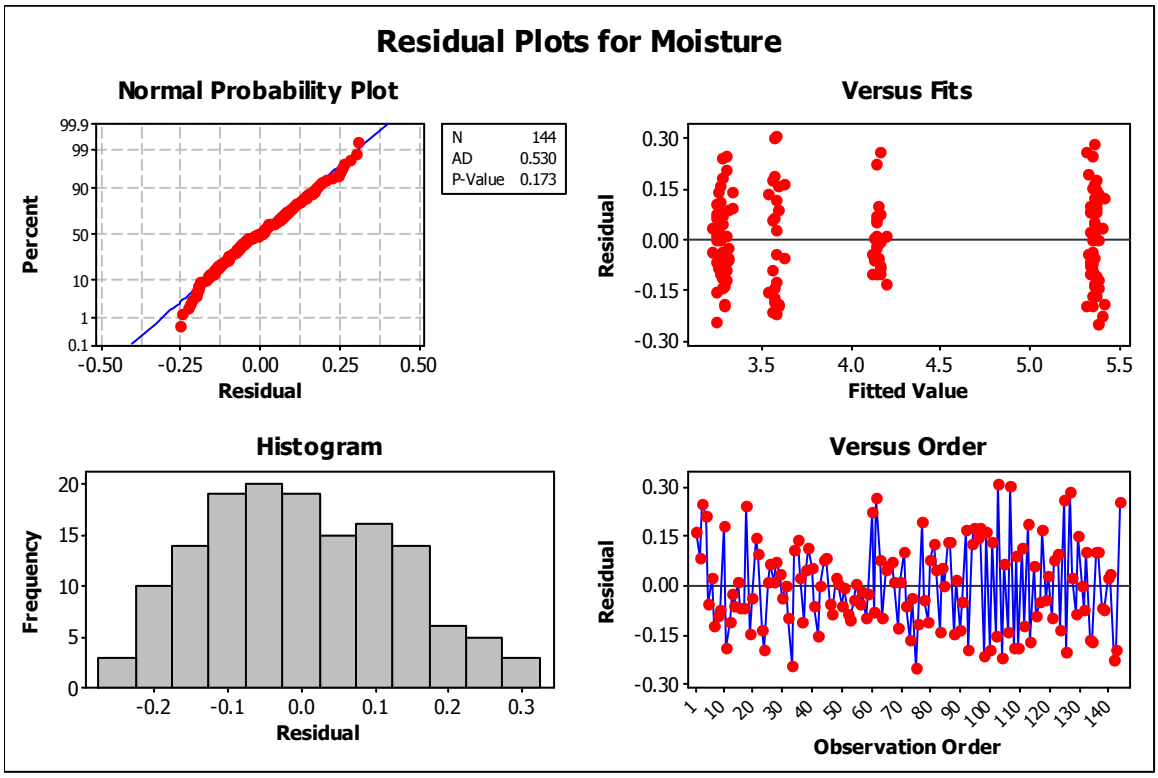

Figure 76: Residual plots for moisture content of powders as determined by drying for 5 h in a vacuum oven at 100ºC; Top Left: Normal probability plot (p = 0.173); Top Right: Residuals versus fitted values; Bottom Left: Histogram of Residuals; Bottom Right: Residuals versus order (Excluding "0 time" data)

Appendix L: Minitab Output for Solubility

One-way ANOVA: BM, NFDM, WMP, MPI, SW, WPC

```
Source  DF        SS       MS  F  P
Factor   5  21.22575  4.24515  *  *
Error   72   0.00000  0.00000
Total   77  21.22575

S = 0   R-Sq = 100.00%   R-Sq(adj) = 100.00%


                              Individual 95% CIs For Mean Based on Pooled StDev
Level   N     Mean    StDev    +---------+---------+---------+---------
BM     13  0.12000  0.00000       *
NFDM   13  0.10000  0.00000       *
WMP    13  0.10000  0.00000       *
MPI    13  1.50000  0.00000                                           *
SW     13  0.00000  0.00000    *
WPC    13  0.25000  0.00000          *
                                +---------+---------+---------+---------
                              0.00      0.40      0.80      1.20

Pooled StDev = 0.00000


Grouping Information Using Tukey Method

       N     Mean  Grouping
MPI   13  1.50000  A
WPC   13  0.25000     B
BM    13  0.12000        C
WMP   13  0.10000           D
NFDM  13  0.10000           D
SW    13  0.00000              E

Means that do not share a letter are significantly different.


Tukey 95% Simultaneous Confidence Intervals
All Pairwise Comparisons

Individual confidence level = 99.54%


BM subtracted from:

         Lower    Center     Upper  ---------+---------+---------+---------+
NFDM  -0.02000  -0.02000  -0.02000                    *
WMP   -0.02000  -0.02000  -0.02000                    *
MPI    1.38000   1.38000   1.38000                                      *
SW    -0.12000  -0.12000  -0.12000                   *
WPC    0.13000   0.13000   0.13000                      *
                                   ---------+---------+---------+---------+
                                         -0.80      0.00      0.80      1.60


NFDM subtracted from:

        Lower    Center     Upper  ---------+---------+---------+---------+
WMP   0.00000   0.00000   0.00000                    *
```

```
MPI   1.40000   1.40000   1.40000                                   *
SW   -0.10000  -0.10000  -0.10000                        *
WPC   0.15000   0.15000   0.15000                           *
                                    ---------+---------+---------+---------+
                                          -0.80      0.00      0.80      1.60
```

WMP subtracted from:

```
        Lower    Center     Upper  ---------+---------+---------+---------+
MPI   1.40000   1.40000   1.40000                                   *
SW   -0.10000  -0.10000  -0.10000                        *
WPC   0.15000   0.15000   0.15000                           *
                                    ---------+---------+---------+---------+
                                          -0.80      0.00      0.80      1.60
```

MPI subtracted from:

```
        Lower    Center     Upper  ---------+---------+---------+---------+
SW   -1.50000  -1.50000  -1.50000  *
WPC  -1.25000  -1.25000  -1.25000     *
                                    ---------+---------+---------+---------+
                                          -0.80      0.00      0.80      1.60
```

SW subtracted from:

```
       Lower   Center    Upper  ---------+---------+---------+---------+
WPC  0.25000  0.25000  0.25000                        *
                                ---------+---------+---------+---------+
                                      -0.80      0.00      0.80      1.60
```